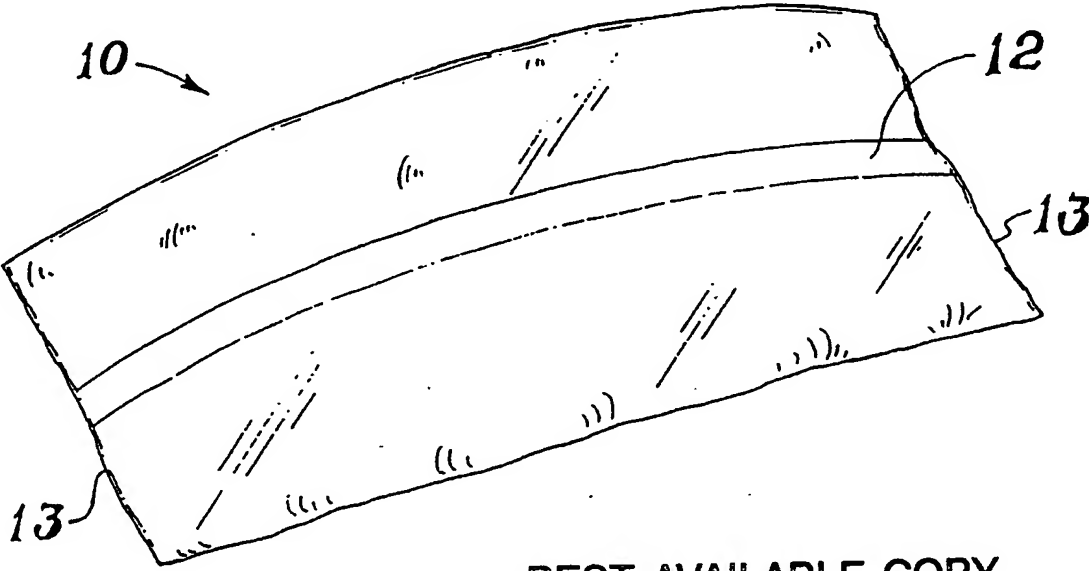




INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 5 : B32B 27/08, C09J 123/04 B65D 65/40	A1	(11) International Publication Number: WO 93/02859 (43) International Publication Date: 18 February 1993 (18.02.93)
(21) International Application Number: PCT/US92/06582 (22) International Filing Date: 7 August 1992 (07.08.92) (30) Priority data: 742,800 9 August 1991 (09.08.91) US (71) Applicant: THE DOW CHEMICAL COMPANY [US/ US]; 2030 Dow Center, Abbott Road, Midland, MI 48640 (US). (72) Inventors: FALLA, Daniel, James ; 1347 Errol Road East, Sarnia, Ontario N7S 5J8 (CA). ELLIOTT, Robert, Scott ; 1343 Cathcart Boulevard, Sarnia, Ontario N7S 2H7 (CA). ROSS, Allen, Ward ; 755 Manor Park, Sarnia, On- tario N7S 4W7 (CA).		(74) Agent: PRIETO, Joe, R.; The Dow Chemical Company, Patent Department, P.O. Box 1967, Midland, MI 48641-1967 (US). (81) Designated States: AU, CA, FI, JP, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, SE). Published <i>With international search report.</i>
(54) Title: POUCH FOR PACKAGING FLOWABLE MATERIALS  BEST AVAILABLE COPY		
(57) Abstract An environmentally friendly polymer film pouch made from a polyethylene film structure for the packaging of flowable materials, for example milk, including, for example, a pouch made from a multilayer film structure such as a two-layer or a three-layer coextruded film containing at least one layer of ultra low density linear polyethylene, as a seal layer.		

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POUCH FOR PACKAGING FLOWABLE MATERIALS

This invention relates to a pouch used in consumer packaging made from certain film structures useful for packaging flowable materials, for example liquids such as milk.

5 U.S. Patent Nos. 4,503,102 and 4,521,437 disclose the preparation of a polyethylene film for use in the manufacture of a disposable pouch for packaging of liquids such as milk. U.S. Patent No. 4,503,102 discloses pouches made from a blend of a linear ethylene copolymer copolymerized from ethylene and an alpha-olefin at the C₄ to C₁₀ range and a ethylene-vinyl acetate polymer copolymerized from ethylene and vinyl acetate. The
10 linear polyethylene copolymer has a density of from 0.916 to 0.930 g/cm³ and a melt index of from 0.3 to 2.0 g/10 minutes. The ethylene-vinyl acetate polymer has a weight ratio of ethylene to vinyl acetate from 2.2:1 to 24:1 and a melt index of from 0.2 to 10 g/10 minutes. The blend disclosed in U.S. Patent No. 4,503,102 has a weight ratio of linear low density polyethylene to ethylene-vinyl acetate polymer of from 1.2:1 to 4:1. U.S. Patent No. 4,503,102 also discloses
15 laminates having as a sealant film the aforementioned blend.

U.S. Patent No. 4,521,437 describes pouches made from a sealant film which is from 50 to 100 parts of a linear copolymer of ethylene and octene-1 having a density of from 0.916 to 0.930 g/cm³ and a melt index of 0.3 to 2.0 g/10 minutes and from 0 to 50 parts by weight of at least one polymer selected from the group consisting of a linear copolymer of
20 ethylene and a C₄-C₁₀-alpha-olefin having a density of from 0.916 to 0.930 g/cm³ and a melt index of from 0.3 to 2.0 g/10 minutes, a high-pressure polyethylene having a density of from 0.916 to 0.924 g/cm³ and a melt index of from 1 to 10 g/10 minutes and blends thereof. The sealant film disclosed in the U.S. Patent No. 4,521,437 is selected on the basis of providing (a) pouches with an M-test value substantially smaller, at the same film thickness, than that
25 obtained for pouches made with film of a blend of 85 parts of a linear ethylene/butene-1 copolymer having a density of about 0.919 g/cm³ and a melt index of about 0.75 g/10 minutes and 15 parts of a high pressure polyethylene having a density of about 0.918 g/cm³ and a melt index of 8.5 g/10 minutes, or (b) an M(2)-test value of less than about 12 percent, for pouches having a volume of from greater than 1.3 to 5 liters, or (c) an M(1.3)-test value of less than

about 5 percent for pouches having a volume of from 0.1 to 1.3 liters. The M, M(2) and M(1.3)-tests are defined pouch drop tests in U.S. Patent No. 4,521,437. The pouches may also be made from composite films in which the sealant film forms at least the inner layer.

The polyethylene pouches known in the prior art have some deficiencies. The
5 problems associated with the prior art known films relate to the sealing properties and performance properties of the film for preparing pouches. In particular, prior art films made into pouches have a high incident of "leakers", i.e., seal defects such as pinholes which develop at or near the seal in which flowable material, for example milk, escapes from the pouch. Although the seal and performance properties of the prior art films have been satisfactory,
10 there is still a need in the industry for better seal and performance properties in films for manufacture of hermetically sealed pouches containing flowable materials. More particularly, there is a need for improved sealing properties of the film such as hot tack and heat seal initiation temperature in order to improve the processability of the film and to improve pouches made from the films.

For example, the line speed of known packaging equipment used for
15 manufacturing pouches such as form, fill and seal machines, is currently limited by the sealing properties of the film used in the machines. Prior art polyethylene films have high hot tack seal initiation temperatures and a narrow sealing range. Therefore, the speed at which a form, fill and seal machine can produce a pouch is limited and, thus, the number of pouches produced
20 on a form, fill and seal machine is limited. If the heat seal temperature range, where one could obtain strong seals, is broadened, then the speed of a form, fill and seal machine can be increased and, thus, the number of pouches produced can be increased. Until the present invention, many have attempted to broaden the heat seal temperature range of pouch film without success.

25 It is desired to provide a polyethylene film structure for a pouch container having a broad heat sealing range with performance properties as good or better than the known prior art pouch films.

It is also desired to provide a film structure for a pouch container having a heat
seal layer of ultra low density polyethylene such that the film structure has a broader sealing
30 range for pouch conversion and has acceptable physical properties in the finished product.

It is further desired to provide a pouch made from the aforementioned film structures such that the pouch has a reduced failure rate.

One aspect of the present invention is directed to a pouch for the packaging of
liquid consumer products, said pouch made from a film structure including at least one layer of
35 an ultra low density polyethylene being a linear ethylene copolymer interpolymerized from ethylene and at least one alpha-olefin in the range of C₃-C₁₀ and having (1) a density of less than about 0.915 g/cm³, (2) a melt index of less than about 10.0 g/10 minutes and (3) (i) a hot tack or heat seal initiation temperature of less than about 100°C at a force of at least about 1

N/inch (39.4 N/m) or (ii) achieving a hot tack strength of at least 1 N/inch (39.4 N/m) at a seal bar temperature of about 110°C and at less than about 0.2 seconds using the DTC Hot Tack Strength Method or achieving a heat seal strength of at least 1 lbf/inch (175 N/m) at a seal bar temperature of about 110°C and at less than about 0.25 seconds using the DTC Heat Seal

5 Strength Method.

One embodiment of the present invention is a pouch made from a two-layer coextruded film containing an outer layer of linear low density polyethylene and an inner seal layer of the aforementioned ultra low density linear polyethylene.

Another embodiment of the present invention is a pouch made from a three-
10 layer coextruded film containing an outer layer and a core layer of linear low density polyethylene and an inner seal layer of the aforementioned ultra low density linear polyethylene.

Another aspect of the present invention is a process for preparing the aforementioned pouch.

15 It has been discovered that the film structures for the pouches of the present invention have a better seal at lower sealing temperatures and shorter dwell times than currently obtainable with commercially available film. Use of the films for making pouches of the present invention in form, fill and seal machines leads to machine speeds higher than currently obtainable with the use of commercially available film.

20 Figure 1 shows a perspective view of a pouch package of the present invention.
Figure 2 shows a perspective view of another pouch package of the present invention.

Figure 3 shows a partial, enlarged cross-sectional view of the film structure of a pouch of the present invention.

25 Figure 4 shows another partial, enlarged cross-sectional view of another embodiment of the film structure of a pouch of the present invention.

Figure 5 shows yet another partial, enlarged cross-sectional view of another embodiment of the film structure of a pouch of the present invention.

30 Figures 6-8 are graphical illustrations of hot tack strength of various film structures versus temperature.

Figures 9-11 are graphical illustrations of hot tack strength of various film structures versus sealing time.

Figures 12-14 are graphical illustrations of heat seal strength of various film structures versus temperature.

35 Figures 15-17 are graphical illustrations of heat seal strength of various film structures versus sealing time.

In its broadest scope, the pouch of the present invention, for example as shown in Figures 1 and 2, for packaging flowable materials is manufactured from a monolayer film

structure of a polymeric seal layer which is a polyethylene film layer, preferably, a polyethylene referred to hereinafter as "ultra low density polyethylene" ("ULDPE"). An example of a commercially available ULDPE is ATTANE™ (Trademark of and commercially available from The Dow Chemical Company). The ULDPE of the present invention is generally a linear copolymer of ethylene with at least one α -olefin having from 3 to 10 carbon atoms, for example, the ULDPE may be selected from ethylene-1-propylene, ethylene-1-butene, ethylene-1-pentene, ethylene-4-methyl-1-pentene, ethylene-1-hexene, ethylene-1-heptene, ethylene-1-octene and ethylene-1-decene copolymers, preferably ethylene-1-octene copolymer.

Generally, the polymeric seal layer has a density of less than about 0.915 g/cm³, preferably from 0.89 to 0.915 g/cm³; generally has a melt index of less than about 10 g/10 minutes, preferably from 0.1 to 10 g/10 minutes; more preferably from 0.5 to 5.0 g/10 minutes; and generally has an indicator of molecular weight distribution (I_{10}/I_2) of less than about 20, preferably from 5 to 20, more preferably from 7 to 20 and even more preferably from 6 to 18. The thickness of the seal layer may be from at least about 0.1 mil (2.5 microns) and greater, preferably from 0.2 mil (5 microns) to 10 mil (254 microns) and more preferably from 0.4 mil (10 microns) to 5 mil (127 microns).

A surprising feature of the pouch's film structure of the present invention is the film's broad heat sealing range. Generally, the heat sealing range of the film structure can be from 70°C to 140°C and preferably from 75°C to 130°C. It has been found that the seal layer of the present invention has a broader heat seal range than prior art polyethylene film having higher densities. A broad heat sealing range is important to allow for more flexibility in the heat sealing process used for making pouches from the film structure.

Another unexpected feature of the pouch's film structure of the present invention is the film's heat seal strength at low temperatures. Generally, the film structure of the present invention achieves a hot tack strength of at least about 1 N/inch (39.4 N/m) at a seal bar temperature of about 110°C and at less than about 0.2 seconds using the DTC Hot Tack Strength Method defined hereinbelow or achieves a heat seal strength of at least 1 lb_f/inch (175 N/m) at a seal bar temperature of about 110°C and at less than 0.25 seconds using the DTC Heat Seal Strength Method defined hereinbelow. The film structure of the present invention also has a hot tack or heat seal initiation temperature of less than about 100°C at a force of at least about 1 N/inch (39.4 N/m). It has been found that a seal made with the seal layer of the present invention has a higher strength at lower sealing temperatures than seals with a prior art polyethylene having higher densities. A high heat seal strength at low temperatures is important to allow conventional packaging equipment such as a vertical form, fill and seal machine to run at faster rates and to produce pouches with fewer leakers.

It is believed that the use of ULDPE in a film structure for pouches of the present invention (1) provides a pouch that can be fabricated at a fast rate through a form, fill and seal machine and (2) provides a pouch package having few leakers, particularly when the pouch of

the present invention is compared to pouches made with linear low density polyethylene, low density polyethylene or a combination thereof.

Another embodiment of the present invention includes a pouch made from a blend of (a) from 10 to 100 percent by weight of at least one linear ethylene copolymer
5 interpolymers from ethylene and at least one alpha-olefin in the range of C₃-C₁₀ and having a density of less than about 0.915 g/cm³ and a melt index of less than about 10.0 g/10 minutes, and (b) from 0 to 90 percent by weight of at least one polymer selected from the group consisting of a linear copolymer of ethylene and a C₃-C₁₈-alpha-olefin having a density of greater than about 0.916 g/cm³ and a melt index of from 0.1 to 10 g/10 minutes, a high-
10 pressure low-density polyethylene having a density of from 0.916 to 0.930 g/cm³ and a melt index of from 0.1 to 10 g/10 minutes and an ethylene-vinyl acetate (EVA) copolymer having a weight ratio of ethylene to vinyl acetate from 22:1 to 24:1 and a melt index of from 0.2 to 20 g/10 minutes.

With reference to Figures 3 to 5, the film structure of the pouch of the present
15 invention also includes a multilayer or composite film structure 30, preferably containing the above-described polymeric seal layer being the inner layer of the pouch.

As will be understood by those skilled in the art, the multilayer film structure for the pouch of the present invention may contain various combination of film layers as long as the seal layer forms part of the ultimate film structure. The multilayer film structure for the
20 pouch of the present invention may be a coextruded film, a coated film or a laminated film. The film structure also includes the seal layer in combination with a barrier film such as polyester, nylon, EVOH, polyvinylidene dichloride (PVDC) such as SARAN™ (Trademark of The Dow Chemical Company) and metallized films. The end use for the pouch tends to dictate, in a large degree, the selection of the other material or materials used in combination with the seal
25 layer film. The pouches described herein will refer to seal layers used at least on the inside of the pouch.

One embodiment of the film structure 30 for the pouch of the present invention, shown in Figure 3, comprises an ultra low density polyethylene seal layer 31 and at least one polymeric outer layer 32. The polymeric outer layer 32 is preferably a polyethylene film layer,
30 more preferably a polyethylene referred to hereinafter as "linear low density polyethylene" ("LLDPE"). An example of a commercially available LLDPE is DOWLEX™ 2073 (Trademark of and commercially available from The Dow Chemical Company). The LLDPE is generally a linear copolymer of ethylene and a minor amount of an alpha-olefin having from 3 to 18 carbon atoms, preferably from 4 to 10 carbon atoms and most preferably 8 carbon atoms. The LLDPE
35 for the outer layer 32 generally has a density of greater than 0.916 g/cm³, more preferably from 0.916 to 0.935 g/cm³, more preferably from 0.918 to 0.926 g/cm³; generally has a melt index of from 0.1 to 10 g/10 minutes, preferably from 0.5 to 2 g/10 minutes; and generally has an I₁₀/I₂

ratio of from 5 to 20, preferably from 7 to 20. The thickness of the outer layer 32 may be any thickness so long as the seal layer 31 has a minimum thickness of about 0.1 mil (2.5 microns).

Another embodiment of the film structure 30 for the pouch of the present invention, shown in Figure 4, comprises the polymeric layer 32 sandwiched between two
5 polymeric seal layers 31.

Still another embodiment of the film structure 30 for the pouch of the present invention, shown in Figure 5, comprises at least one polymeric core layer 33 between at least one polymeric outer layer 32 and at least one polymeric seal layer 31. The polymeric layer 33 may be the same LLDPE film layer as the outer layer 32 or preferably a different LLDPE, and
10 more preferably an LLDPE, for example DOWLEX™ 2049 (Trademark of and commercially available from The Dow Chemical Company) that has a higher density than the outer layer 32. The thickness of the core layer 33 may be any thickness so long as the seal layer 31 has a minimum thickness of about 0.1 mil (2.5 microns).

Yet another embodiment (not shown) of the film structure for the pouch of the
15 present invention can be a structure including a seal layer 31 and another polyethylene film layer referred to hereinafter as "high pressure low-density polyethylene" ("LDPE"). The LDPE layer generally has a density of from 0.916 to 0.930 g/cm³ and has a melt index of from 0.1 to 10 g/10 minutes. The thickness of the LDPE layer may be any thickness so long as the seal layer 31 has a minimum thickness of about 0.1 mil (2.5 microns).

20 Still another embodiment (not shown) of the film structure for the pouch of the present invention can be a structure including a seal layer 31 and a layer of EVA copolymer having a weight ratio of ethylene to vinyl acetate from 22:1 to 24:1 and a melt index of from 0.2 to 20 g/10 minutes. The thickness of the EVA layer may be any thickness so long as the seal layer 31 has a minimum thickness of about 0.1 mil (2.5 microns).

25 The ultimate film thickness of the final film product used for making the pouch of the present invention is from 0.5 mil (12.7 microns) to 10 mils (254 microns), preferably from 1 mil (25.4 microns) to 5 mils (127 microns); more preferably from 2 mils (50.8 microns) to 4 mils (100 microns).

Additives, known to those skilled in the art, such as anti-block agents, slip
30 additives, UV stabilizers, pigments and processing aids may be added to the polymers from which the pouches of the present invention are made.

As can be seen from the different embodiments of the present invention shown in Figures 3-5, the film structure for the pouches of the present invention has design flexibility. Different LLDPE can be used in the outer and core layers to optimize specific film properties
35 such as film stiffness. Thus, the film can be optimized for specific applications such as for a vertical form, film and seal machine.

The polyethylene film structure used to make a pouch of the present invention is made by either the blown tube extrusion method or the cast extrusion method, methods well

known in the art. The blown tube extrusion method is described, for example, in Modern Plastics Mid-October 1989 Encyclopedia Issue, Volume 66, Number 11, pages 264 to 266. The cast extrusion method is described, for example, in Modern Plastics Mid-October 1989 Encyclopedia Issue, Volume 66, Number 11, pages 256 to 257.

5 Embodiments of the pouches of the present invention, shown in Figures 1 and 2, are hermetically sealed containers filled with "flowable materials". By "flowable materials" it is meant, materials which are flowable under gravity or which may be pumped, but the term "flowable materials" does not include gaseous materials. The flowable materials include liquids for example milk, water, fruit juice, oil; emulsions for example ice cream mix, soft
10 margarine; pastes for example meat pastes, peanut butter; preservers for example jams, pie fillings marmalade; jellies; doughs; ground meat for example sausage meat; powders for example gelatin powders, detergents; granular solids for example nuts, sugar; and like materials. The pouch of the present invention is particularly useful for liquid foods for example milk. The flowable material may also include oleaginous liquids for example, cooking oil or
15 motor oil.

 Once the film structure for the pouch of the present invention is made, the film structure is cut to the desired width for use in conventional pouch-forming machines. The embodiments of the pouch of the present invention shown in Figures 1 and 2 are made in so-called form, fill and seal machines well known in the art. With regard to Figure 1, there is
20 shown a pouch 10 being a tubular member 11 having a longitudinal lap seal 12 and transverse seals 13 such that, a "pillow-shaped" pouch is formed when the pouch is filled with flowable material.

 With regard to Figure 2, there is shown a pouch 20 being a tubular member 21 having a peripheral fin seal 22 along three sides of the tubular member 11, that is, the top seal
25 22a and the longitudinal side seals 22b and 22c, and having a bottom substantially concave or "bowl-shaped" member 23 sealed to the bottom portion of the tubular seal 21 such that when viewed in cross-section, longitudinally, substantially a semi-circular or "bowed-shaped" bottom portion is formed when the pouch is filled with flowable material. The pouch shown in Figure 2 is the so-called "Enviro-Pak" pouch known in the art.

30 The pouch manufactured according to the present invention is preferably the pouch shown in Figure 1 made on so-called vertical form, fill and seal (VFFS) machines well known in the art. Examples of commercially available VFFS machines include those manufactured by Hayssen or Prepac. A VFFS machine is described in the following reference: F. C. Lewis, "Form-Fill-Seal," Packaging Encyclopedia, page 180, 1980.

35 In a VFFS packaging process, a sheet of the plastic film structure described herein is fed into a VFFS machine where the sheet is formed into a continuous tube in a tube-forming section. The tubular member is formed by sealing the longitudinal edges of the film together -- either by lapping the plastic film and sealing the film using an inside/outside seal or by fin

sealing the plastic film using an inside/inside seal. Next, a sealing bar seals the tube transversely at one end being the bottom of the "pouch", and then the fill material, for example milk, is added to the "pouch." The sealing bar then seals the top end of the pouch and either burns through the plastic film or cuts the film, thus, separating the formed completed pouch from the tube. The process of making a pouch with a VFFS machine is generally described in U.S. Patent Nos. 4,503,102 and 4,521,437 incorporated herein by reference.

The capacity of the pouches of the present invention may vary. Generally, the pouches may contain from 5 milliliters to 10 liters, preferably from 1 milliliter to 8 liters, and more preferably from 1 milliliter to 5 liters of flowable material.

The film structure for the pouch of the present invention has precisely controlled strength. The use of the film structure described in the present invention for making a pouch results in a stronger pouch, and, therefore, more preferably, the pouch contains fewer use-related leakers. The use of the ULDPE seal layer of the present invention in a two or three-layer coextruded film product will provide a film structure that can be used for making pouches at a faster rate in the VFFS and such pouches produced will contain fewer leakers.

The pouches of the present invention have excellent performance results when tested by the Milk Pouch Drop Test and Step Stair Drop Test--tests which are defined herein. Under the Step Stair Drop Test, the pouches preferably have a "50 percent failure height" of greater than about 10 feet (3.0 m) and more preferably greater than about 13 feet (4 m). Under the Milk Pouch Drop Test, the pouches of the present invention preferably have a failure rate of less than about 10 percent and more preferably less than about 7 percent.

With the trend in today's consumer packaging industry moving toward providing the consumer with more environmentally friendly packages, the polyethylene pouch of the present invention is a good alternative. The use of the polyethylene pouch for packaging consumer liquids such as milk has its advantages over containers used in the past: the glass bottle, paper carton, and high density polyethylene jug. The previously used containers consumed large amounts of natural resources in their manufacture, required a significant amount of space in landfill, used a large amount of storage space and used more energy in temperature control of the product (due to the heat transfer properties of the container).

The polyethylene pouch of the present invention made of thin polyethylene film, used for liquid packaging, offers many advantages over the containers used in the past. The polyethylene pouch (1) consumes less natural resources, (2) requires less space in a landfill, (3) can be recycled, (4) can be processed easily, (5) requires less storage space, (6) uses less energy for storage (heat transfer properties of package), (7) can be safely incinerated and (8) can be reused, for example, the empty pouch can be used for other applications such as freezer bags, sandwich bags, and general purpose storage bags.

The following resins described in Table I were used to make blended and coextruded blown film samples described in the Examples and Comparative Examples:

Table I

Resin Designation	Resin Type	Melt Index (g/10 minutes)	Density (g/cm ³)
Resin A	LDPE	2.0	0.922
Resin B	LLDPE	0.75	0.921
Resin C	LLDPE	1.0	0.926
Resin D	ULDPE	1.0	0.912
Resin E	ULDPE	0.8	0.905

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Erucamide, a slip agent; SiO₂, an antiblock agent; and a processing aid were added to each of the resins described in Table I such that the final concentrations of the additives were as follows: 1200 ppm Erucamide; 2500 ppm SiO₂ (4000 ppm for the ULDPE coextruded products); and 900 ppm processing aid. Film structures were made at 2 mil (50.8 microns) and 3 mil (76.2 microns) target thickness.

15

Film structures produced were subjected to physical testing to determine its various properties including:

- (1) Puncture, using method ASTM D3763;
- (2) Dart Impact, using ASTM D1709, Method A and Method B;
- (3) Elmendorf Tear, using ASTM D1922;
- 20 (4) Tensiles, using ASTM D882;
- (5) Gloss, using method ASTM D2457;
- (6) Clarity, using method ASTM D1746;
- (7) Coefficient of Friction, using method ASTM D1894;
- (8) 2 percent Secant Modulus, using ASTM D882;
- 25 (9) Hot Tack Strength (only 3 mil (76.2 microns) films), using method described hereinbelow; and
- (10) Heat Seal Strength (only 3 mil (76.2 microns) films), using method described hereinbelow;

30 The present invention is illustrated by the following examples but is not to be limited thereby.

Examples 1-4 and Comparative Examples A and B

Film samples described in Table II were made as a monolayer using a Macro blown film line. The extruder was 2-1/2 inches (6.4 cm) in diameter and had a 24:1 L/D and a barrier screw with a Maddock mixing head. A 6 inch (15.2 cm) diameter die was used with a 40 mil
35 (1,016 microns) die gap for the manufacture of the test films.

Table II: Monolayer Films

Example No.	Sample No.	Resin	Gauge (mil)
1	Sample 1A	Blend of Resin B (75%)/Resin D (25%)	3
2	Sample 1B	Blend of Resin B (75%)/Resin D (25%)	2
3	Sample 2A	Blend of Resin C (75%)/Resin D (25%)	3
4	Sample 2B	Blend of Resin C (75%)/Resin D (25%)	2
Comparative A	Comparative Sample 1A	Resin B	3
Comparative B	Comparative Sample 1B	Resin B	2

The monolayer films shown in Table II were produced in the Macro blown film line using the following fabrication conditions:

2.5 blow-up ratio

216°C melt temperature

5 The results of testing the above film samples are shown in Tables VI and VII.

Examples 5-24 and Comparative Examples C and D

The film samples described in Tables III, IV and V were produced using an Egan 3-layer coextrusion line.

10 The fabrication conditions used in the production of the coextruded films was as follows:

2.5 blow-up ratio

216°C melt temperature

The results of testing the above films samples are shown in Tables VI and VII.

15 Comparative films samples, Samples 2A and 2B, described in Table V were produced and tested using the same conditions and equipment as in Example 5-24 except that Comparative Samples 2A and 2B were blends and not multilayer coextruded films. The results of testing the comparative film samples are shown in Tables VI and VII.

Comparative Example E

20 Comparative Sample 3 in Table VI is SCLAIRFILM SM-3 film (herein SM-3 Film) commercially available from DuPont Canada. Two 24-centimeters diameter x 38 centimeters x 3 mil (76.2 microns) rolls of SM-3 Film were tested concurrently with the fabricated films of the present invention as in Examples 5-24. A publication by DuPont Canada discloses that SM-3 Film has a density of 0.918 g/cm³.

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Table III: 2-Layer Coextrusion

Example No.	Sample No.	Extruder A Inner	Extruder B Core	Extruder C Outer	Gauge (mil)
5	Sample 3A	Resin D (50%)	Resin B (25%)	Resin B (25%)	3
6	Sample 3B	Resin D (50%)	Resin B (25%)	Resin B (25%)	2
7	Sample 4A	Resin D (25%)	Resin B (37.5%)	Resin B (37.5%)	3
8	Sample 4B	Resin D (25%)	Resin B (37.5%)	Resin B (37.5%)	2
9	Sample 5A	Resin D (50%)	Resin C (25%)	Resin C (25%)	3
10	Sample 5B	Resin D (50%)	Resin C (25%)	Resin C (25%)	2
11	Sample 6A	Resin D (25%)	Resin C (37%)	Resin C (37%)	3
12	Sample 6B	Resin D (25%)	Resin C (37%)	Resin C (37%)	2

Table IV: 3-Layer Coextrusion

Example No.	Sample No.	Extruder A Inner	Extruder B Core	Extruder C Outer	Gauge (mil)
13	Sample 7A	Resin D (20%)	Resin C (60%)	Resin B (20%)	3
14	Sample 7B	Resin D (20%)	Resin B (60%)	Resin B (20%)	2
15	Sample 8A	Resin D (33%)	Resin C (33%)	Resin B (33%)	3
16	Sample 8B	Resin D (33%)	Resin C (33%)	Resin B (33%)	2
17	Sample 9A	Resin E (20%)	Resin C (60%)	Resin B (20%)	3
18	Sample 9B	Resin E (20%)	Resin C (60%)	Resin B (20%)	2
19	Sample 10A	Resin E (33%)	Resin C (33%)	Resin B (33%)	3
20	Sample 10B	Resin E (33%)	Resin C (33%)	Resin B (33%)	2
21	Sample 11A	Resin D (20%)	Resin B (60%)	Resin D (20%)	3
22	Sample 11B	Resin D (20%)	Resin B (60%)	Resin D (20%)	2
23	Sample 12A	Resin D (33%)	Resin B (33%)	Resin D (33%)	3
24	Sample 12B	Resin D (33%)	Resin B (33%)	Resin D (33%)	2

Table V: Blended Films

Example No.	Sample No.	<u>Extruder A</u> Inner	<u>Extruder B</u> Core	<u>Extruder C</u> Outer	Gauge (mil)
Comparative C	Comparative Sample 2A	Blend of Resin B (85%)/ Resin A (15%)	Blend of Resin B (85%)/ Resin A (15%)	Blend of Resin B (85%)/ Resin A (15%)	3
Comparative D	Comparative Sample 2B	Blend of Resin B (85%)/ Resin A (15%)	Blend of Resin B (85%)/ Resin A (15%)	Blend of Resin B (85%)/ Resin A (15%)	2

Table VI: Film Performance Data, 3 mil

Property	Monolayer Structure				2-Layer Structure			
	1A	2A	Comparative 1A	Comparative 2A	3A	4A	5A	6A
Gauge, mil (microns)								
Average	3.18 (80.8)	3.09 (78.5)	3.23 (82.0)	3.24 (82.3)	3.28 (83.3)	3.23 (82.0)	3.29 (83.6)	2.96 (75.2)
Range	2.93-3.45 (74.4-87.6)	2.84-3.39 (72.1-86.1)	2.98-3.69 (75.7-93.7)	3.18-3.35 (80.8-85.1)	3.20-3.39 (81.3-86.1)	3.11-3.45 (79.0-87.6)	3.05-3.51 (77.5-89.2)	2.90-3.02 (73.7-76.7)
Coefficient of Friction								
Static:								
inside/outside	0.22	0.28	0.25	0.26	0.22	0.24	0.26	0.25
outside/outside					0.24	0.22		
inside/inside					0.24	0.23		
Kinetic:								
inside/outside	0.17	0.18	0.18	0.19	0.19	0.18	0.20	0.20
outside/outside					0.20	0.19		
inside/inside					0.17	0.17		
Clarity, percent	26.2	37.3	31.5	11.6	13.7	24.1	30.4	36.6
Gloss, percent	63.9	62.6	60.9	62.2	62.5	62.1	60.6	61.6
Elmendorf Tear, g	1,539	1,234	1,391	1,175	1,638	1,595	1,480	1,146
MD ⁽¹⁾	2,001	1,687	1,900	1,911	1,862	1,818	1,731	1,549
CD ⁽²⁾								
Dart Impact, g	492		374	420	375	452	362	420
Large Dart	584	459	516		516	603	519	
Small Dart								

Table VI: Film Performance Data, 3 mil (cont.)

Property	Monolayer Structure				2-Layer Structure			
	1A	2A	Comparative 1A	Comparative 2A	3A	4A	5A	6A
2% Secant, psi (MPa)	23947 (1651.1)	29377 (2025.5)	26647 (1837.3)	25847 (1782.2)	20477 (1411.9)	22731 (1567.3)	25620 (1766.5)	29740 (2050.6)
Puncture, ft-lbf/in ³ (J/cm ³)	27379 (1887.8)	33096 (2282.0)	30668 (2114.6)	27811 (1917.6)	23428 (1615.4)	27146 (1871.7)	29915 (2062.6)	34202 (2358.2)
	16.2 (1.34)	16.8 (1.39)	16.7 (1.38)	13.3 (1.10)	12.8 (1.06)	13.7 (1.13)	13.3 (1.10)	15.2 (1.26)
Tensile Yield, psi (MPa)	1461 (100.70)	1680 (115.84)	1633 (112.58)	1543 (106.38)	1319 (90.93)	1428 (98.46)	1525 (105.11)	1737 (119.79)
Ultimate, psi (MPa)	1562 (107.67)	1865 (128.58)	1827 (126.00)	1581 (109.02)	1405 (96.85)	1552 (107.03)	1726 (118.98)	1929 (133.03)
	5676 (391.37)	5655 (389.88)	5713 (393.93)	4908 (338.41)	5456 (376.19)	5656 (389.99)	6042 (416.60)	5953 (410.48)
Elongation, percent	5698 (392.86)	5314 (366.42)	5366 (370.01)	4610 (317.86)	5497 (379.02)	5711 (393.77)	5987 (412.77)	5603 (386.30)
	692 (753)	965 (974)	963 (751)	914 (974)	944 (1004)	936 (999)	1004 (1029)	978 (987)
Toughness Modulus, ft-lbf/in ³ (J/cm ³)	1360 (112.4)	2018 (166.9)	2163 (178.9)	1778 (147.0)	1774 (146.7)	1831 (151.4)	2103 (173.9)	2104 (174.0)
	1500 (124.1)	1969 (162.9)	1574 (130.2)	1753 (145.0)	1876 (155.2)	1962 (162.2)	2184 (180.6)	2072 (171.4)

Table VI: Film Performance Data, 3 mil (cont.)

Property	3-Layer Structure						Comparative Sample 3*
	7A	8A	9A	10A	11A	12A	
Gauge, mil (microns)							
Average	3.22 (81.8)	2.93 (74.4)	2.83 (71.9)	2.95 (74.9)	3.39 (86.1)	3.31 (84.1)	3.17 (80.5)
Range	3.18-3.35 (80.8-85.1)	2.68-3.06 (68.1-77.7)	2.77-2.93 (70.4-74.4)	2.89-3.02 (73.4-76.7)	3.10-3.55 (78.7-90.2)	3.25-3.37 (82.9-85.6)	2.90-3.34 (73.7-84.8)
Coefficient of Friction							
Static:							
inside/outside	0.22	0.22	0.24	0.27	0.25	0.25	0.13
outside/outside	0.23	0.21	0.23	0.25	0.24	0.27	0.12
inside/inside	0.22	0.18	0.29	0.28	0.23	0.24	0.14
Kinetic:							
inside/outside	0.18	0.17	0.19	0.21	0.18	0.20	0.10
outside/outside	0.17	0.17	0.20	0.22	0.19	0.21	0.10
inside/inside	0.16	0.15	0.21	0.24	0.17	0.18	0.10
Clarity, percent	32.2	27.6	35.0	31.2	31.0	25.7	45.1
Gloss, percent	65.4	69.2	65.4	66.3	69.2	69.4	74.1
Elmendorf Tear, g	1,285	1,402	1,184	1,354	1,798	1,711	758
MD ⁽¹⁾	1,690	1,578	1,585	1,651	1,997	1,943	1,248
CD ⁽²⁾							
Dart Impact, g							
Large Dart	462	459	399	372	529	621	366
Small Dart				492	>651	>651	465

Table VI: Film Performance Data, 3 mil (cont.)

Property	3-Layer Structure						Comparative Sample 3*
	7A	8A	9A	10A	11A	12A	
2% Secant, psi (MPa)	27431 (1891.4)	23098 (1592.6)	25428 (1753.3)	23439 (1616.1)	24874 (1715.1)	22373 (1542.6)	22800 (1572.1)
CD(2)	30348 (2092.5)	26283 (1812.2)	31921 (2201.6)	30288 (2088.4)	29083 (2005.3)	26883 (1853.6)	25449 (1754.7)
Puncture, ft-lbf/in ³ (J/cm ³)	14.2 (1.17)	12.3 (1.02)	15.8 (1.31)	16.6 (1.37)	13.1 (1.08)	13.4 (1.11)	13.8 (1.14)
Tensile	1.664 (114.74)	1.463 (100.85)	1.575 (108.58)	1.486 (102.48)	1.428 (98.44)	1.374 (94.73)	1.558 (107.43)
Yield, psi (MPa)	1.837 (126.64)	1.608 (110.86)	1.763 (121.55)	1.617 (111.47)	1.623 (111.89)	1.506 (103.82)	1.524 (105.07)
Ultimate, psi (MPa)	5.953 (410.45)	5.348 (368.77)	5.194 (358.13)	5.882 (405.58)	6.008 (414.26)	5.908 (407.37)	5.639 (388.82)
CD	5.844 (402.95)	4.718 (325.27)	5.089 (350.91)	5.748 (396.31)	5.892 (406.28)	5.866 (404.44)	6.173 (425.65)
Elongation, percent	989 (1.016)	924 (937)	927 (956)	927 (995)	998 (1,037)	971 (1,026)	761 (682)
Toughness Modulus, ft-lbf/in ³ (J/cm ³)	2.083 (172.2)	1.750 (144.7)	1.759 (145.4)	1.869 (154.5)	2.039 (168.7)	1.951 (161.3)	1.562 (129.1)
CD	2.130 (176.1)	1.665 (137.7)	1.819 (150.4)	2.000 (165.4)	2.144 (177.3)	2.046 (169.2)	1.569 (129.8)

1)MD = machine direction

(2)CD = cross direction

*SM-3 Film Commercially available from DuPont Canada

Table VII: Film Performance Data, 2 mil

Property	Monolayer Structure				2-Layer Structure			
	1B	2B	Comparative 1B	Comparative 2B	3B	4B	5B	6B
Gauge, mil (micron)	2.11 (53.6)	2.08 (52.8)	2.01 (51.1)	2.14 (54.4)	2.11 (53.6)	1.97 (50.0)	2.08 (52.8)	2.00 (50.8)
Average								
Range	1.88-2.3 (47.8-58.9)	1.91-2.42 (48.5-61.5)	1.78-2.18 (45.2-55.4)	2.06-2.34 (52.3-59.4)	1.92-2.15 (48.8-54.6)	1.90-2.01 (48.3-51.1)	2.04-2.13 (51.8-54.1)	1.91-2.05 (48.5-52)
Clarity, percent	27.0	40.1	26.5	17.7	22.5	28.1	32.6	34.7
Gloss, percent	64.7	63.1	54.5	64.7	67.7	67.4	60.1	59.5
Elmendorf Tear, g	963	817	832	653	956	882	945	707
MD(1)								
CD(2)	1,341	1,174	1,277	1,363	1,235	1,148	1,162	1,094
Dart Impact, Small Dart, g	465	317	327	273	405	417	339	294
2% Secant, psi (MPa)	24276 (1673.6)	27506 (1896.3)	27088 (1867.4)	26483 (1825.7)	20396 (1406.1)	22959 (1582.8)	25582 (1763.6)	30418 (2097.0)
CD	27077 (1866.7)	32326 (2228.6)	31375 (2163.0)	29419 (2028.1)	23421 (1614.6)	27135 (1870.7)	30813 (2124.2)	36867 (2541.6)
Puncture, ft-lb/in ³ (J/cm ³)	16.3 (1.34)	18.7 (1.55)	19.3 (1.60)	10.6 (0.88)	15.4 (1.27)	15.2 (1.26)	14.0 (1.16)	13.7 (1.13)

Table VII: Film Performance Data, 2 mil (cont.)

Property	Monolayer Structure				2-Layer Structure			
	1B	2B	Comparative 1B	Comparative 2B	3B	4B	5B	6B
Tensile Yield, psi (MPa)	MD(1)	1,492 (102.87)	1,619 (111.63)	1,671 (115.16)	1,583 (109.10)	1,288 (88.77)	1,408 (97.10)	1,546 (106.55)
	CD(2)	1,550 (106.85)	1,804 (124.37)	1,746 (120.37)	1,705 (117.53)	1,369 (96.23)	1,490 (102.71)	1,671 (115.18)
Ultimate, psi (MPa)	MD	5,710 (393.62)	5,779 (398.40)	5,588 (385.26)	5,138 (354.23)	5,403 (372.51)	5,345 (368.47)	5,804 (400.11)
	CD	4,775 (329.17)	4,919 (339.10)	4,164 (287.04)	4,975 (342.99)	4,647 (320.37)	4,775 (329.17)	4,945 (340.92)
Elongation, percent	MD	843	651	625	824	859	822	910
	CD	926	666	663	956	911	897	936
Toughness Modulus, ft-lb/in ³ (J/cm ³)	MD	1,682 (1139.1)	1,316 (108.8)	1,293 (106.9)	1,731 (143.2)	1,580 (130.6)	1,525 (126.1)	1,857 (153.6)
	CD	1,728 (142.9)	1,249 (103.3)	1,149 (95.0)	1,837 (151.9)	1,548 (128.0)	1,549 (128.1)	1,737 (143.6)

Table VII: Film Performance Data, 2 mil (cont.)

Property	3-Layer Structure					
	7B	8B	9B	10B	11B	12B
Gauge, mil (micron)						
Average	2.08 (52.8)	2.12 (53.8)	2.09 (53.1)	2.04 (51.8)	2.14 (54.4)	2.21 (56.1)
Range	2.02-2.17 (51.3-55.1)	2.09-2.14 (53.1-54.4)	2.07-2.14 (52.6-54.4)	2.01-2.08 (51.1-52.8)	2.02-2.27 (51.3-57.7)	2.01-2.29 (51.1-58.2)
Clarity, percent						
Gloss, percent	34.1	28.2	35.0	34.6	27.5	26.2
Elmendorf Tear, g	70.2	72.2	70.9	66.3	67.3	69.4
MD(1)	896	915	851	787	1,030	1,157
CD(2)	1,220	1,112	1,079	1,143	1,267	1,385
Dart Impact, Small Dart, g	315	345	285	390	483	519
2% Secant, psi (MPa)	27421 (1890.4)	23854 (1644.5)	29096 (2005.9)	25967 (1790.2)	23163 (1569.9)	22977 (1584.0)
MD	29076	28520	32290	30064	27064	25982
CD	(2004.5)	(1966.2)	(2226.1)	(2072.6)	(1865.8)	(1791.2)

Table VII: Film Performance Data, 2 mil (cont.)

Property	3-Layer Structure					
	7B	8B	9B	10B	11B	12B
Puncture, ft-lbf/in ³ (J/cm ³)	16.7 (1.38)	15.6 (1.29)	17.5 (1.45)	18.7 (1.55)	14.7 (1.22)	14.3 (1.18)
Tensile						
Yield, psi (MPa)	1,569 (108.15)	1,515 (104.44)	1,575 (108.59)	1,508 (103.95)	1,427 (98.36)	1,374 (94.71)
	1,848 (127.40)	1,628 (112.21)	1,681 (115.87)	1,589 (109.53)	1,576 (108.66)	1,456 (100.40)
Ultimate, psi (MPa)	5,557 (383.07)	5,006 (345.11)	4,721 (325.49)	5,371 (370.30)	5,546 (382.33)	5,632 (388.28)
	5,036 (347.17)	4,532 (312.42)	4,616 (318.25)	5,194 (358.07)	5,209 (359.07)	4,977 (343.14)
Elongation, percent	916 919	857 907	805 900	815 914	895 939	874 949
Toughness Modulus, ft-lbf/in ³ (J/cm ³)	1,803 (149.1)	1,581 (130.8)	1,436 (118.7)	1,539 (127.3)	1,721 (149.5)	1,695 (70.1)
	1,795 (148.4)	1,596 (132.0)	1,582 (130.9)	1,697 (140.4)	1,766 (146.6)	1,696 (0.0)

(1)MD = machine direction

(2)CD = cross direction

In liquid packaging, the pouches may be subjected to a wide variety of abuses. These include dropping of the pouch on the floor, dropping objects onto the pouch, picking the pouch up from one end and poking it with fingers or other objects. Performance Tests--puncture, dart drop test, Elmendorf tear and tensile--are intended to duplicate the type of abuse that the pouches would encounter during normal use. In general, the properties of the film structure for the pouches of the present invention were as good, and in some cases better than, the properties of prior art films.

For example, in the puncture test, of the 3 mil (76.2 microns) films that were tested, Sample 2A, Comparative Sample 1A and Sample 10A had the highest puncture resistance. Of the 2-mil (50.8 microns) gauge films, Comparative Sample 1B had the highest puncture resistance, followed by Sample 10B.

In the Elmendorf Tear test, however, it was unexpectedly found that some prior art films performed poorly in the Elmendorf tear tests, while the coextruded films with ULDPE in one of the layers performed well. The films made with LLDPE either coextruded or monolayer, typically had tear values less than the coextruded films with ULDPE in one of the layer.

The coefficient of friction (COF) property of the films generally ranged from 0.10 to 0.30.

In order for a film to properly move over the forming collars in a vertical form, fill and seal machine, the film is required to have a specific COF range. If the COF is high, the film may be too tacky for the VFFS to pull the film over the forming collar. If the COF is low, the film may be too slippery and the pull belts may not be able to grip the film to pull it. The process of coextrusion advantageously allows for varying slip properties between the inside and outside of the film by varying the slip concentration in the independent film layers.

The "2 percent secant modulus" property of the film structures is a measure of film stiffness. The stiffness of the film structures (2 percent secant modulus) is measured according to the method of ASTM-D882.

A specific amount of stiffness in a film is required for use of the film for producing pouches. If too much stiffness is present in the film, the film could experience too much fold when pulled over the edge of the forming collar and forming tube of a VFFS. Excessive stiffness can cause the film to "hang-up" in the VFFS. On the other hand, if enough stiffness is not present in the film consumer problems are inevitable. For example, in milk packaging, a pouch is usually placed inside of a support container which holds the pouch upright with approximately 2-1/2 inches (6 cm) to 3-1/2 inches (9 cm) of the pouch remaining above the container. To open the pouch, the corner is cut with a pair of scissors. If the film does not have enough stiffness (or wall strength), the film wall could collapse while the consumer is pouring the liquid from the pouch.

The film structures for the pouches of the present invention advantageously have precisely controlled stiffness which is required for the film structure to run through a VFFS. Generally, the stiffness of the film structure of the present invention is from 1,400 MPa machine direction (MD)/1600 MPa cross direction (CD) to 2,100 MPa MD/2,500 MPa CD and preferably from 1,412 MPa MD/1,615 MPa CD to 2,050 MPa MD/2,358 MPa CD. If the film lacks stiffness, the film may become "bunched" in the corners of the VFFS unit. If the film is too stiff, the film will not bend properly for the sealing of longitudinal edges.

Advantageously, the stiffness of the structure can be changed by using different polyethylene layers. For example, it was found that by varying the amount of the higher density resin, for example Resin C (LLDPE having a density of 0.926) used in the core layer of the coextruded films, the stiffness of the film could be altered. For example, film Sample 7A had a MD 2 percent secant value of 1,891 MPa and film Sample 8A had a MD 2 percent secant value of 1,593 MPa.

The above Examples illustrates that the use of ULDPE as the sealing layer in a film structure for the pouch of the present invention allows for the development of a designed structure with the appropriate amount of tear resistance, dart impact resistance, elongation and stiffness (2 percent secant).

Example 25 - Hot Tack Strength

The hot tack strength of the 3 mil (76.2 microns) films was measured using the "DTC Hot Tack Test Method." The "DTC Hot Tack Test Method" is a test method which measures the force required to separate a heat seal before the seal has had a chance to fully cool (crystallize). This simulates the filling of material into a pouch before the seal has had a chance to cool.

The "DTC Hot Tack Test Method" is a test method using a DTC Hot Tack Tester Model #52D according to the following conditions:

	Specimen Width:	25.4 mm
	Sealing Time:	0.5 seconds
	Sealing Pressure:	0.27 N/mm/mm
30	Delay Time:	0.5 seconds
	Peel Speed:	150 mm/second
	Number of Samples/Temperature:	3
	Temperature Increments:	5°C
35	Temperature Range:	70°C - 130°C

The hot tack results for the individual films may be found in Table VIII.

Table VIII: Hot Tack Seal Initiation Temperature and Hot Tack Strength

5	Sample No.	Hot Tack Seal Initiation Temperature (°C)	Temperature at Maximum Hot Tack Strength (°C)	Maximum Hot Tack Strength, N/inch (N/m)
	Sample 1A	98	120	3.3 (130)
	Sample 2A	105	120	3.1 (122)
10	Comparative Sample 1A	102.5	115	3.4 (134)
	Comparative Sample 2A	102	115	3.5 (138)
	Sample 3A	86	115	3.2 (126)
	Sample 4A	86.5	115	3.4 (134)
15	Sample 5A	87	115	2.9 (114)
	Sample 6A	88.5	105	3.1 (122)
	Sample 7A	86.5	105	3.7 (146)
	Sample 8A	86	105	3.3 (130)
	Sample 9A	76.5	105	3.4 (134)
20	Sample 10A	76.5	105	3.3 (130)
	Sample 11A	87	105	3.4 (134)
	Sample 12A	87	105	3.2 (126)
	Comparative Sample 3	104.5	120	3.6 (142)

25 The "Maximum Hot Tack Strength" (maximum hot tack seal force) of the films and the temperature at which the Maximum Hot Tack Strength of the films occurs is shown in Table VIII.

The "Hot Tack Seal Initiation Temperature" ("Hot Tack T_i ") shown in Table VIII is the lowest temperature at which a seal is formed. A seal force of 1.0 N/inch (39.4 N/m) was
 30 selected as the force required to form an adequate seal, and therefore, Hot Tack T_i is found at a force of 1.0 N/inch (39.4 N/m).

A low Hot Tack T_i and a broad heat seal range is important for VFFS packaging. A low initiation temperature and a broad heat seal range allows the VFFS machine to run at faster line speeds by allowing the sealing jaws of the VFFS to close for short periods of time
 35 while still obtaining an adequate heat seal.

The Hot Tack T_i for 3 mil (76.2 microns) films in Table VIII shows that the coextruded films with Resin E in the seal layer showed the lowest Hot Tack T_i (76.5°C) followed by films with Resin D in the sealing layer (86°C).

The 3-layer and 2-layer coextruded films with an ULDPE in the sealing layer had the lowest temperature (105°C) at which the highest hot tack strength was achieved.

Figures 6-8 illustrate the Hot Tack Seal Initiation Temperature and the temperature at which the maximum Hot Tack Strength was achieved for various film samples.

- 5 The temperature between Hot Tack T_i and the temperature of maximum Hot Tack Strength indicates the size of the hot tack sealing range. Figures 6-8 shows that films with ULDPE as the sealing layer have a much larger sealing range than the LLDPE and/or LLDPE/LDPE blend films.

Example 26 - Hot Tack Strength versus Sealing Time

- 10 This Example was carried out using the DTC Hot Tack Test Method described in Example 25 except that the temperature was held constant at 110°C and the sealing time was varied from 0.1 second to 1 second. Only 3 mil (76.2 microns) films were tested.

The conditions used on the DTC Hot Tack Tester Model #52D were as follows:

	Specimen Width:	25.4 mm
	Sealing Time:	varied
15	Delay Pressure:	0.27 N/mm/mm
	Peel Speed:	150 mm/second
	Number of Samples/Time:	3
	Sealing Time Range:	0.1 second to 1 second
	Temperature:	110°C

- 20 The results of the Hot Tack Strength for the various films are found in Table IX.
Table IX

Seal Time (s)	Sample Number Hot Tack Strength, N/inch (N/m)						
	Compara- tive Sample 2A	Compara- tive Sample 3	Sample 1A	Sample 3A	Sample 8A	Sample 10A	Sample 12A
0.0							
0.1	0.17 (6.7)	0.30 (11.8)	0.68 (26.8)	2.07 (81.6)	2.21 (87.1)	2.92 (115)	2.37 (93.4)
0.2	0.58 (22.9)	0.85 (33.5)	1.74 (69.6)	2.75 (108)	3.03 (119)	3.04 (120)	2.89 (114)
0.4	1.89 (74.5)	1.46 (70.9)	2.46 (96.9)	2.89 (114)	3.07 (121)	3.06 (121)	3.26 (128)
0.6	2.40 (94.6)	1.80 (70.9)	2.52 (99.3)	3.06 (121)	3.23 (127)	3.12 (123)	3.15 (124)
0.8	2.23 (87.9)	1.97 (77.6)	2.57 (101)	3.17 (125)	3.32 (131)	3.30 (130)	3.28 (129)
1.0	2.36 (93.0)	1.99 (78.4)	2.55 (100)	3.15 (124)	3.39 (134)	3.39 (134)	3.25 (128)

Tables VIII and IX show that films made with ULDPE in the sealing layer have higher hot tack strengths for shorter sealing times than the comparative samples tested at 110°C.

The data of Table IX is shown specifically in Figures 9 to 11.

5 Example 27 - Heat Seal Strength

The heat seal strength of the 3 mil (76.2 microns) films was measured using the "DTC Heat Seal Strength Test Method." The "Heat Seal Strength Test Method" is a test method which measures the force required to separate a seal after the material has cooled to 23°C temperature. The film samples were exposed to a relative humidity of 50 percent and a
10 temperature of 23°C for a minimum of 24 hours prior to testing.

The "DTC Heat Seal Strength Test Method" is a test method using a DTC Hot Tack Tester Model #52D, wherein the heat seal portion of the tester is used, according to the following conditions:

15	Specimen Width:	25.4 mm
	Sealing Time:	0.5 seconds
	Sealing Pressure:	0.27 N/mm/mm
	Number of Samples/Temperature:	3
20	Temperature Increments:	10°C
	Temperature Range:	70°C - 140°C

The seal strength of the film samples was determined using an Instron Tensile Tester Model #1122 according to the following test conditions:

25	Direction of Pull:	90° to seal
	Crosshead Speed:	500 mm/minute
	Full Scale Load:	5 kg
	Number of Samples/Threshold:	31 percent of FSL
30	Break Criterion:	80 percent
	Gauge Length:	2.0 inches (50.8 millimeters)
	Sample Width:	1.0 inch (25.4 millimeters)

35 The monolayer films were tested inside/inside, while the coextruded films were tested inside/inside and inside/outside.

The heat seal results for the individual films are found in Table X.

Table X: Heat Seal Initiation Temperature and Maximum Heat Seal Strength

Sample No.	Inside/Inside		Inside/Outside			
	Initiation Temperature (°C)	Temperature at Maximum Heat Seal Strength (°C)	Maximum Heat Seal Strength, lbf/inch (N/m)	Initiation Temperature (°C)	Temperature at Maximum Heat Seal Strength (°C)	Maximum Heat Seal Strength, lbf/inch (N/m)
Sample 1A	104	130	6.33 (1,108)			
Sample 2A	103.5	130	6.26 (1,096)			
Comparative Sample 1A	109.5	130	6.30 (1,103)			
Comparative Sample 2A	110	140	7.91 (1,385)			
Sample 3A	95	140	5.87 (1,028)	105.5	140	6.57 (1,151)
Sample 4A	104.5	140	6.01 (1,053)	106.5	130	6.56 (1,149)
Sample 5A	95.5	130	5.92 (1,037)	106	130	5.94 (1,040)
Sample 6A	95.5	130	6.12 (1,012)	110	140	6.68 (1,170)
Sample 7A	94	130	6.40 (1,121)	103.5	140	6.32 (1,107)
Sample 8A	96.5	130	6.04 (1,058)	102	140	6.45 (1,130)
Sample 9A	82.5	90	5.89 (1,032)	102	140	4.99 (874)
Sample 10A	82.5	90	6.35 (1,112)	85.5	130	4.64 (813)
Sample 11A	97	130	6.27 (1,098)			
Sample 12A	101.5	130	6.17 (1,081)			
Comparative Sample 3	103.5	140	8.20 (1,436)			

The "Maximum Heat Seal Strength" (maximum heat seal force) of the film samples and the temperature at which the Maximum Heat Seal Strength of the films occurs is shown in Table X.

The "Heat Seal Initiation Temperature" ("Heat Seal T_i ") shown in Table X is the lowest temperature at which a seal is formed. A seal force of 1.0 lbf/inch (175 N/m) was selected as the force required to form an adequate seal, and therefore, Heat Seal T_i is found at a force of 1.0 lb/in (175 N/m).

A low Heat Seal T_i and a broad heat seal range is important for VFFS packaging. A low initiation temperature and a broad heat seal range allows the VFFS machine to run at faster line speeds by allowing the sealing jaws of the VFFS to close for short periods of time while still obtaining an adequate heat seal.

The monolayer films and most of the coextruded films have a smooth heat seal curve as shown in Figures 12-14; however, the films with higher concentrations of ULDPE in the sealing layer appeared to have two maximum sealing temperatures. It is believed that these two peaks are the maximum sealing forces achieved for the ULDPE layer and the LLDPE layer. As the heat is applied to the film, the ULDPE layer begins to form a seal at lower temperatures than the LLDPE (Heat Seal T_i of 82.5°C vs. 111°C). As more heat is applied to the seal, the ULDPE layer reaches its maximum sealing temperature and strength. This phenomenon occurs below the Heat Seal T_i for the LLDPE film. After the maximum sealing temperature has been achieved, the sealing strength drops and then it increases reaching a maximum at the same temperature that the LLDPE film does.

As expected, based on the hot tack results, the films with Resin E in the sealing layer had the lowest Heat Seal T_i and the LLDPE had the highest Heat Seal T_i . The Heat Seal T_i results were very similar to the Hot Tack T_i results.

25 Example 28 - Heat Seal Strength versus Sealing Time

This Example was carried out using the DTC Hot Seal Test Method described in Example 27 except that the temperature was held constant at 110°C and the sealing time was varied from 0.1 second to 1 second. Only 3 mil (76.2 microns) films were tested.

The heat seal portion of the DTC Hot Tack #52D Tester Model was used. The conditions on the DTC Hot Tack Tester Model #52D were as follows:

30	Specimen Width:	25.4 mm
	Sealing Time:	varied
	Sealing Pressure:	0.27 N/mm/mm
	Number of Samples/Time:	4
35	Sealing Time Range:	0.1 second to 1 second
	Temperature:	110°C

The seal strength was determined using an Instron Tensile Tester Model No. 1122. The film samples were exposed to a relative humidity of 50 percent and a temperature of 23°C for a minimum of 24 hours prior to testing. The following were the test conditions:

	Direction of Pull:	90° to seal
5	Crosshead Speed:	500 mm/minute
	Full Scale Load:	5 kg
	Threshold:	1 percent of FSL
	Break Criterion:	80 percent
	Gauge Length:	2.0 inches (50.8 mm)
10	Sample Width:	1.0 inch (25.4 mm)

The results of the heat seal force for various films are found in Table XI.

Table XI

Seal Time (s)	Sample Number Heat Seal Strength, lbf/inch (N/m)						
	Compara- tive Sample 2A	Compara- tive Sample 3	Sample 1A	Sample 3A	Sample 8A	Sample 10A	Sample 12A
0.0							
0.1	0.00 (0.00)	0.00 (0.00)	0.12 (21)	1.46 (256)	3.10 (543)	3.68 (644)	2.73 (478)
0.2	0.18 (31)	0.00 (0.00)	0.44 (77)	2.44 (427)	3.49 (611)	3.62 (634)	3.83 (671)
0.4	0.46 (81)	0.25 (44)	2.67 (468)	3.21 (562)	3.57 (625)	3.71 (650)	3.96 (694)
0.6	0.68 (119)	0.32 (56)		3.11 (545)	3.34 (585)	3.41 (597)	4.02 (704)
0.8	0.73 (128)	0.35 (61)	3.82 (669)	3.98 (697)	3.38 (592)	3.49 (611)	3.92 (687)
1.0	0.87 (152)	0.37 (65)	3.84 (672)	3.64 (637)	3.62 (634)	3.19 (559)	3.86 (676)

Examples 25-28 illustrate that the use of ULDPE in the sealing layer of a film structure of the present invention is found to significantly increase the heat seal and hot tack range. The wider range of heat seal and hot tack would allow for faster line speeds on a VFFS unit.

Example 29

A. Pouch Fabrication

A Hayssen Ultima VFFS unit was used to make 2L water-filled pouches with a lay flat dimension of 7 inches (17.8 centimeters) x 12.5 inches (31.8 centimeters) for drop testing.

The following conditions were used on the Hayssen Ultima VFFS unit:

Model Number: RCMB2-PRA

M.A. Number: U19644

Mass of Water: 2,000 g

Bag Size: 7 inches x 12.5 inches

5 Registration rolls on from 5° to 135°

Pull belts on from 5° to 135°

Jaw Close: from 136° to 275°

Platen: from 136° to 265°

Start Delay: 50 ms

10 Type of Seal: Lap

A Pro/Fill 3000 liquid filler was attached to the VFFS. The settings on the Pro/Fill 3000 were:

P.S.: 99

Volume: 3539

15 C.O.A.: 70

B. Drop Testing of Water Filled Pouches

Two types of pouch drop tests were used to measure the performance of films produced in the Examples: (1) Milk Pouch Drop Test, and (2) Step Stair Drop Test. In the Milk Pouch Drop Test pouches were dropped "end-on" from a height of 5 feet (1.5 m). Any leak was
20 classified as a failure. The pouches in the Step Stair Drop Test were also dropped "end-on" from varying heights to determine a pouch's "50 percent failure height". The "50 percent failure height" means the height at which 50 percent of the pouches dropped will fail (leak). Results of the drop testing are shown in Table XII below.

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Table XII: Pouch Drop Tests

	Sample No.	Gauge (mil)	Milk Pouch Drop Test At 5 Feet (1.5 m) Height			Step Stair Drop Test	
			Number Tested	Number Failed	Failure (Percent)	50% Failure Height, Feet (m)	Failure Rate at 13 Feet (4m) (percent)
5	Sample 1A	3	100	7	7	>13 (>4 m)	36
	Sample 1B	2				9.6 (2.88 m)	
	Sample 2A	3	COULD NOT RUN THROUGH VFFS				
	Sample 2B	2	COULD NOT MAKE - POUCH/SEAL FAILURE				
	Comparative Sample 1A	3	COULD NOT MAKE - POUCH/SEAL FAILURE				
10	Comparative Sample 1B	2	COULD NOT MAKE - POUCH/SEAL FAILURE				
	Comparative Sample 2A	3	100	8	8	11.9 (3.57 m)	
	Comparative Sample 2B	2	100	15	15	6.4 (1.92 m)	
	Sample 3A	3				>13 (>4 m)	24
	Sample 3B	2				11.7 (3.51 m)	
15	Sample 4A	3	105	1	0.95	>13 (>4 m)	20
	Sample 4B	2	100	2	2	10.8 (3.24 m)	
	Sample 5A	3				>13 (>4 m)	36
	Sample 5B	2					
	Sample 6A	3	105	1	0.95	>13 (>4 m)	36
20	Sample 6B	2	100	3	3	10.4 (3.13 m)	
	Sample 7A	3	105	1	0.95	>13 (>4 m)	24
	Sample 7B	2	100	2	2	9.8 (2.94 m)	
	Sample 8A	3	100	1	1	>13 (>4 m)	16
	Sample 8B	2				9.2 (2.76 m)	
25	Sample 9A	3	105	0	0	>13 (>4 m)	16
	Sample 9B	2	100	2	2	10.3 (3.09 m)	
	Sample 10A	3	105	0	0	>13 (>4 m)	16
	Sample 10B	2				10.5 (3.15 m)	
	Sample 11A	3	105	1	0.95	>13 (>4 m)	24
30	Sample 11B	2					
	Sample 12A	3				>13 (>4 m)	24
	Sample 12B	2					
	Comparative Sample 3	3	100	7	7	9.1 (2.73 m)	

Film Sample 7A was placed in the VFFS and 130 pouches were made of the sample film at the following temperatures:

Seam seal - 235°F
 Front jaw - 285°F
 Rear jaw - 285°F

One hundred five (105) pouches were dropped as per the Milk Pouch Drop Test and 25 were dropped as per the Step Stair Drop Test. In the Step Stair Drop Test, the maximum height that the pouches could be safely dropped from was 13 feet (4 m) as the results show in Table XII.

The following temperature settings were used to make all of the other films tested and shown in Table XII:

Seam seal - 240°F

Front jaw - 300°F

Rear jaw - 300°F

The number of pouches tested by the Milk Pouch Drop Test is shown in Table XII.

- 5 The number of pouches tested by the Step Stair Drop Test was 25 for all samples.

Most of leakers for the 3 mil (76.2 microns) film in the Milk Pouch Drop Test and Step Stair Drop Test were from the seals. However, most of the leakers for the 2 mil (50.8 microns) films were film failures. The 2 mil (50.8 microns) films appeared to have stronger seals which was expected due to the thinner gauge through which sealing would occur.

- 10 As described in Table XII, the results of the Milk Pouch Drop Test showed that the 3 mil (76.2 microns) films (Samples of 9A and 10A) made with ULDPE (Resin E) in the sealing layer did not have any failures from the 5 foot (1.5 m) drop height. As noted in Table XII, a number of films could not be run on the VFFS.

- Pouches made coextruded films with ULDPE (Resin D) in the sealing layer had Milk
15 Pouch Drop Test failure rates less than the SM-3 Film and LLDPE/LDPE pouches.

- The Milk Pouch Drop Test for the 2 mil (50.8 microns) samples showed that the LLDPE/LDPE pouch (Comparative Sample 2B) had the highest failure rate (15 percent) and Sample 4B with ULDPE layer had the lowest failure rate (2 percent). Unlike the 3 mil (76.2 microns) film where all of the failures were seal related, the 2 mil (50.8 microns) film failures
20 were film related.

With regard to the Step Stair Drop Test, the SM-3 Film was found to have a "50 percent failure height" of about 9.1 feet (2.73 m) as shown in Table XII.

- The 3 mil (76.2 microns) films made with ULDPE had "50 percent failure heights" greater than 13 feet (4 m). Since 13 feet (4 m) was the maximum height that the pouches could
25 be safely dropped, 25 pouch samples were dropped from a height of 13 feet (4 m) and the percent failure at 13 feet (4 m) was determined.

The 2 mil (50.8 microns) LLDPE/LDPE pouch (Comparative Sample 2B) had the lowest "50 percent failure height", while the ULDPE coextruded films had higher "50 percent failure heights". All of the film failures were film related as opposed to seal related.

- 30 Example 29 shows that water filled pouches made with ULDPE in the sealing layer are found to have a low failure rate in the Milk Pouch Drop Test, and a high "50 percent failure height" in the Step Stair Drop Test.

- More particularly, the coextruded films for the pouches of the present invention having a heat seal layer comprised of an ULDPE provide a broader sealing range for pouch
35 conversion and provide physical properties in finished pouches such that the pouches have a reduced failure rate.

CLAIMS:

1. A pouch containing a flowable material, said pouch being made from a film in tubular form and having transversely heat sealed ends, said film being made from a material comprising a film structure comprising (a) from 10 to 100 percent by weight of at least one layer of a linear ethylene copolymer interpolymersized from ethylene and at least one alpha-olefin in the range of C₃-C₁₀ and having a density of less than about 0.915 g/cm³ and a melt index of less than about 10.0 g/10 minutes (i) a hot tack or heat seal initiation temperature of less than about 100°C at a force of at least about 1 N/inch (39.4 N/m) or (ii) achieving a hot tack strength of at least 1 N/inch (39.4 N/m) at a seal bar temperature of about 110°C and at less than about 0.2 seconds using the DTC Hot Tack Strength Method or achieving a heat seal strength of at least 1 lbf/inch (175 N/m) at a seal bar temperature of about 110°C and at less than about 0.25 seconds using the DTC Heat Seal Strength Method; and (b) from 0 to 90 percent by weight of at least one polymer selected from the group consisting of a linear copolymer of ethylene and a C₃-C₁₈-alpha-olefin having a density of greater than about 0.916 g/cm³ and a melt index of from 0.1 to 10 g/10 minutes, a high-pressure low density polyethylene having a density of from 0.916 to 0.930 g/cm³ and a melt index of from 0.1 to 10 g/10 minutes and ethylene-vinyl acetate copolymer having a weight ratio of ethylene to vinyl acetate from 2.2:1 to 24:1 and a melt index of from 0.2 to 10 g/10 minutes.
2. A pouch containing a flowable material, said pouch being made from a multilayer film structure comprising:
- (I) (a) from 10 to 100 percent by weight of at least one layer of a linear ethylene copolymer interpolymersized from ethylene and at least one alpha-olefin in the range of C₃-C₁₀ and having a density of less than about 0.915 g/cm³ and a melt index of less than about 10.0 g/10 minutes (i) a hot tack or heat seal initiation temperature of less than about 100°C at a force of at least about 1 N/inch (39.4 N/m) or (ii) achieving a hot tack strength of at least 1 N/inch (39.4 N/m) at a seal bar temperature of about 110°C and at less than about 0.2 seconds using the DTC Hot Tack Strength Method or achieving a heat seal strength of at least 1 lbf/inch (175 N/m) at a seal bar temperature of about 110°C and at less than about 0.25 seconds using the

DTC Heat Seal Strength Method; and (b) from 0 to 90 percent by weight of at least one polymer selected from the group consisting of a linear copolymer of ethylene and a C₃-C₁₈-alpha-olefin having a density of greater than about 0.916 g/cm³ and a melt index of from 0.1 to 10 g/10 minutes, a high-pressure low density polyethylene having a density of from 0.916 to 0.930 g/cm³ and a melt index of from 0.1 to 10 g/10 minutes and ethylene-vinyl acetate copolymer having a weight ratio of ethylene to vinyl acetate from 2.2:1 to 24:1 and a melt index of from 0.2 to 10 g/10 minutes; and

(II) at least one layer of a linear low density ethylene-C₃-C₁₈-alpha-olefin copolymer having a density of from 0.916 to 0.935 and a melt index of from 0.1 to 10 g/10 minutes.

3. The pouch of Claim 2 comprising:

(III) a high-pressure polyethylene having a density of from 0.916 to 0.930 g/cm³ and a melt index of from 0.1 to 10 g/10 minutes.

4. The pouch of Claim 3 wherein layer (II) is an outer layer, layer (III) is a core layer and layer (I) is a seal layer.

5. The pouch of Claim 1 wherein the pouch holds from 5 mL to 5000 mL of flowable material and the flowable material is milk.

6. The pouch of Claim 1 wherein the copolymer (a) has an indicator of molecular weight distribution (I_{10}/I_2) of from 0.1 to 20.

7. The pouch of Claim 1 wherein the film structure contains a slip agent, antiblock agent, a processing aid, a pigment of an ultraviolet light absorbing additive.

8. A process for preparing a pouch containing a flowable material comprising forming a tubular member having transversely heat-sealed ends, said tubular member being made from a film structure comprising a film structure for a pouch container comprising (a) from 10 to 100 percent by weight of at least one layer of a linear ethylene copolymer interpolymers from ethylene and at least one alpha-olefin in the range of C₃-C₁₀ and having (1) a density of less than about 0.915 g/cm³, (2) a melt index of less than about 10.0 g/10 minutes and (3) (i) a hot tack or heat seal initiation temperature of less than about 100°C at a force of at least about 1 N/inch (39.4 N/m) or (ii) achieving a hot tack strength of at least 1 N/inch (39.4 N/m) at a seal bar temperature of about 110°C and at less than about 0.2 seconds using the DTC Hot Tack Strength Method or achieving a heat seal strength of at least 1 lbf/inch (175 N/m) at a seal bar temperature of about 110°C and at less than about 0.25 seconds using the DTC Heat Seal Strength Method; and (b) from 0 to 90 percent by weight of at least one polymer selected from the group consisting of a linear copolymer of ethylene and a C₃-C₁₈-alpha-olefin having a density of greater than about 0.916 g/cm³ and a melt index of from 0.1 to 10 g/10 minutes, a high-pressure low density polyethylene having a density of from 0.916 to 0.930 g/cm³ and a melt index of from 0.1 to 10 g/10 minutes and ethylene-vinyl acetate

copolymer having a weight ratio of ethylene to vinyl acetate from 2.2:1 to 24:1 and a melt index of from 0.2 to 10 g/10 minutes.

9. A process for preparing a pouch containing a flowable material comprising forming a tubular member having transversely heat-sealed ends, said tubular member being
5 made from a film structure comprising:

(I) (a) from 10 to 100 percent by weight of at least one layer of a linear ethylene copolymer interpolymers from ethylene and at least one alpha-olefin in the range of C₃-C₁₀ and having a density of less than about 0.915 g/cm³ and a melt index of less than about 10.0 g/10 minutes and (i) a hot tack or heat seal initiation temperature of less than about 100°C at a
10 force of at least about 1 N/inch (39.4 N/m) or (ii) achieving a hot tack strength of at least 1 N/inch (39.4 N/m) at a seal bar temperature of about 110°C and at less than about 0.2 seconds using the DTC Hot Tack Strength Method or achieving a heat seal strength of at least 1 lbf/inch (175 N/m) at a seal bar temperature of about 110°C and at less than about 0.25 seconds using the DTC Heat Seal Strength Method; and (b) from 0 to 90 percent by weight of at least one
15 polymer selected from the group consisting of a linear copolymer, of ethylene and a C₃-C₁₈-alpha-olefin having a density of greater than about 0.916 g/cm³ and a melt index of from 0.1 to 10 g/10 minutes, a high-pressure low density polyethylene having a density of from 0.916 to 0.930 g/cm³ and a melt index of from 0.1 to 10 g/10 minutes and ethylene-vinyl acetate copolymer having a weight ratio of ethylene to vinyl acetate from 2.2:1 to 24:1 and a melt
20 index of from 0.2 to 10 g/10 minutes; and

(II) at least one layer of a linear low density ethylene-C₃-C₁₈-alpha-olefin copolymer having a density of from 0.916 to 0.935 and a melt index of from 0.1 to 10 g/10 minutes.

10. The process of Claim 9 wherein the film structure includes:

- 25 (III) a high-pressure polyethylene having a density of from 0.916 to 0.930 g/cm³ and a melt index of from 0.1 to 10 g/10 minutes.

11. A film structure for a pouch container comprising (a) from 10 to 100 percent by weight of at least one layer of a linear ethylene copolymer interpolymers from ethylene and at least one alpha-olefin in the range of C₃-C₁₀ and having (1) a density of less than about
30 0.915 g/cm³, (2) a melt index of less than about 10.0 g/10 minutes and (3) (i) a hot tack or heat seal initiation temperature of less than about 100°C at a force of at least about 1 N/inch (39.4 N/m) or (ii) achieving a hot tack strength of at least 1 N/inch (39.4 N/m) at a seal bar temperature of about 110°C and at less than about 0.2 seconds using the DTC Hot Tack Strength Method or achieving a heat seal strength of at least 1 lbf/inch (175 N/m) at a seal bar
35 temperature of about 110°C and at less than about 0.25 seconds using the DTC Heat Seal Strength Method; and (b) from 0 to 90 percent by weight of at least one polymer selected from the group consisting of a linear copolymer of ethylene and a C₃-C₁₈-alpha-olefin having a density of greater than about 0.916 g/cm³ and a melt index of from 0.1 to 10 g/10 minutes, a

high-pressure low density polyethylene having a density of from 0.916 to 0.930 g/cm³ and a melt index of from 0.1 to 10 g/10 minutes and ethylene-vinyl acetate copolymer having a weight ratio of ethylene to vinyl acetate from 2.2:1 to 24:1 and a melt index of from 0.2 to 10 g/10 minutes.

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Fig. 1

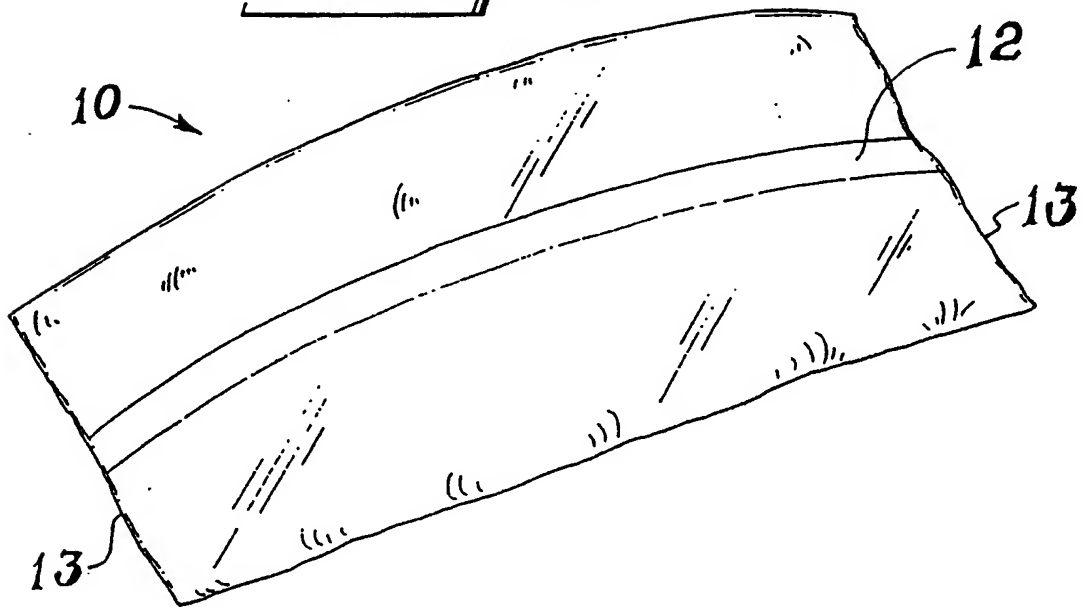
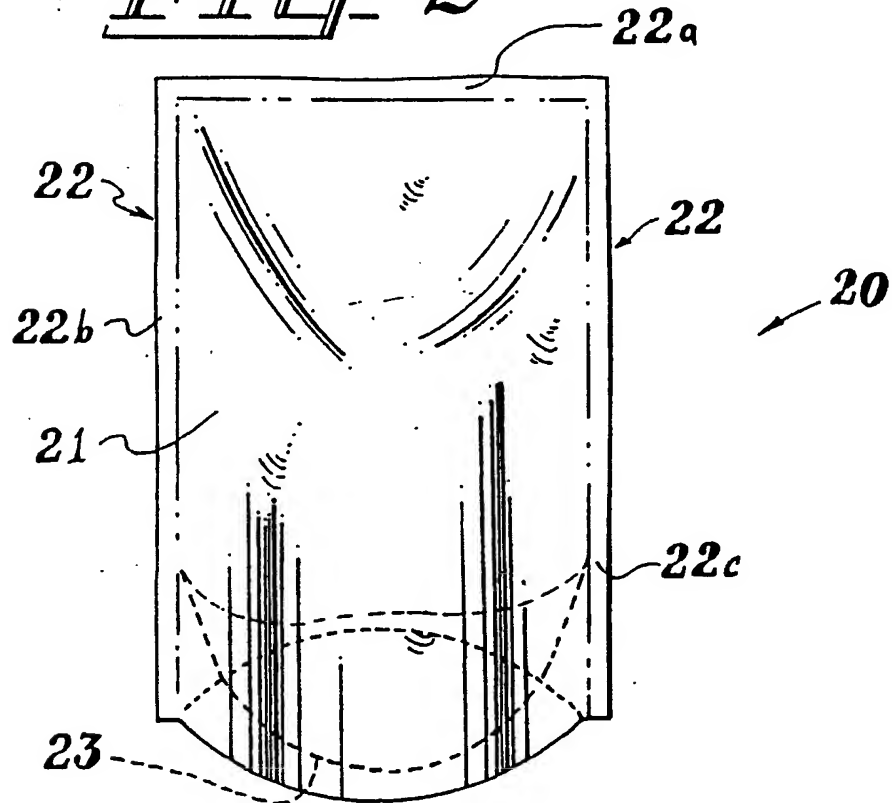


Fig. 2



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FIG. 3

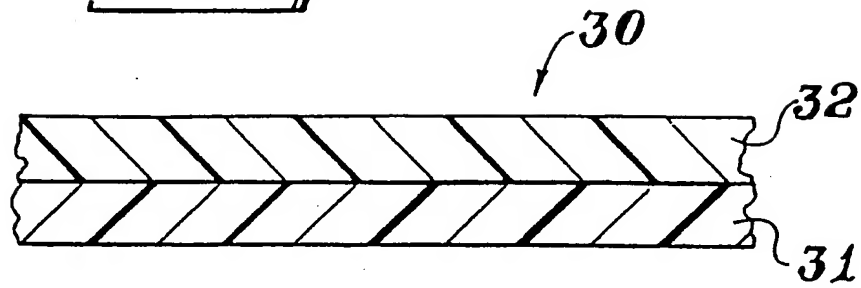


FIG. 4

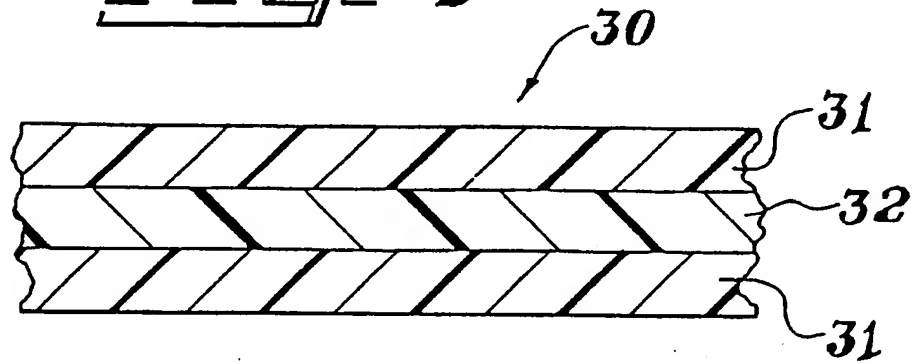
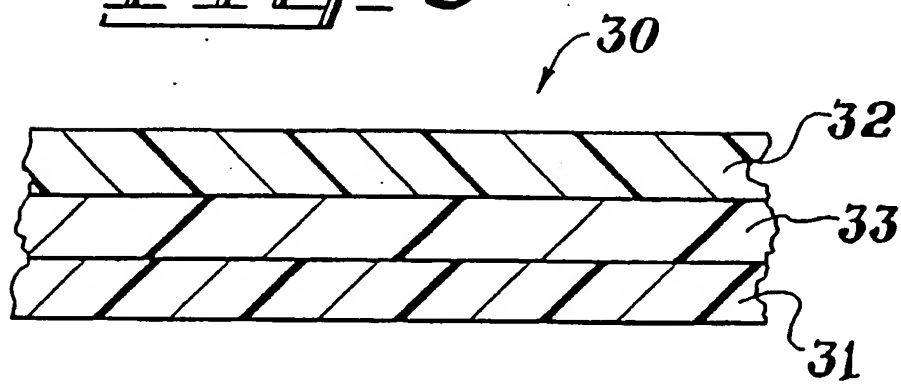


FIG. 5



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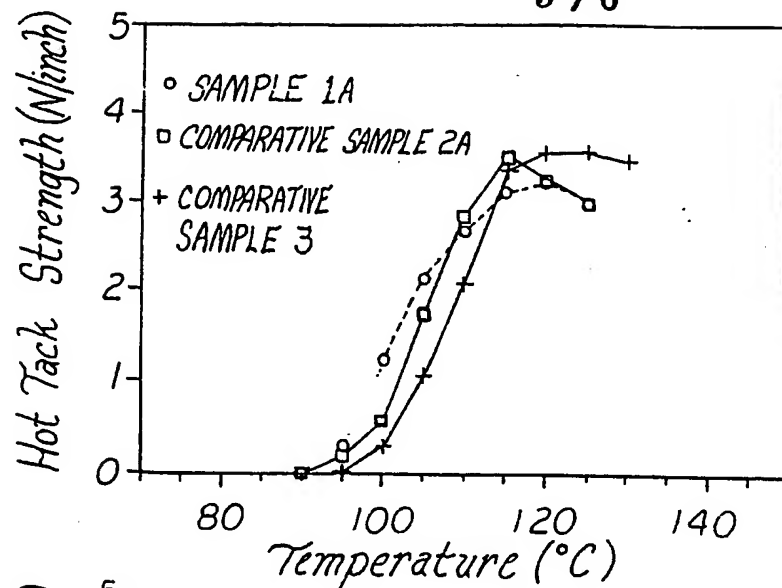


Fig. 6

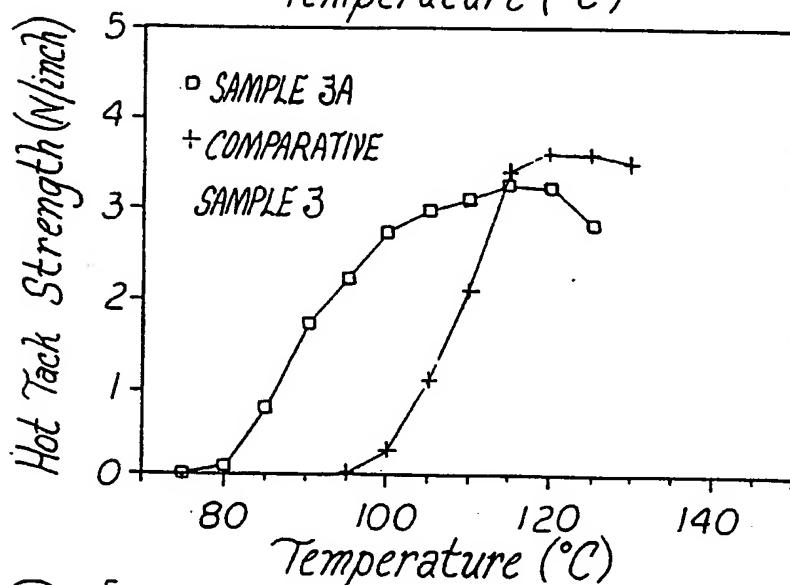


Fig. 7

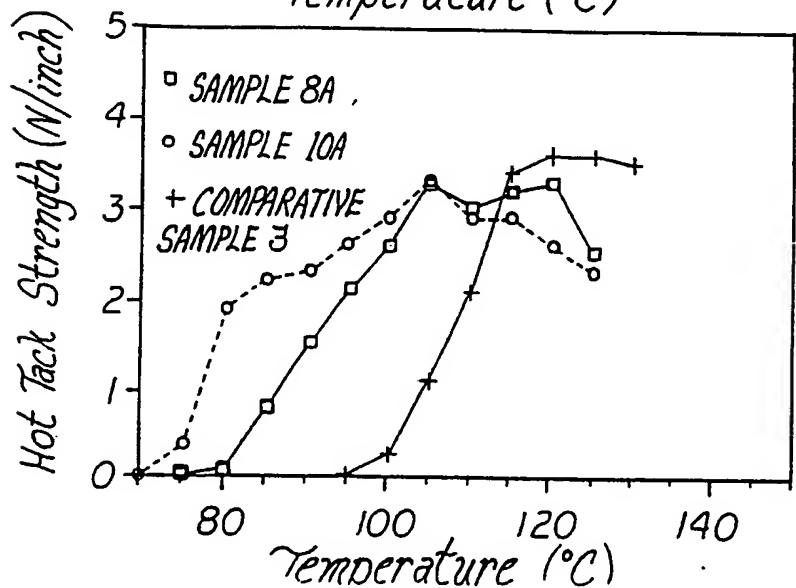
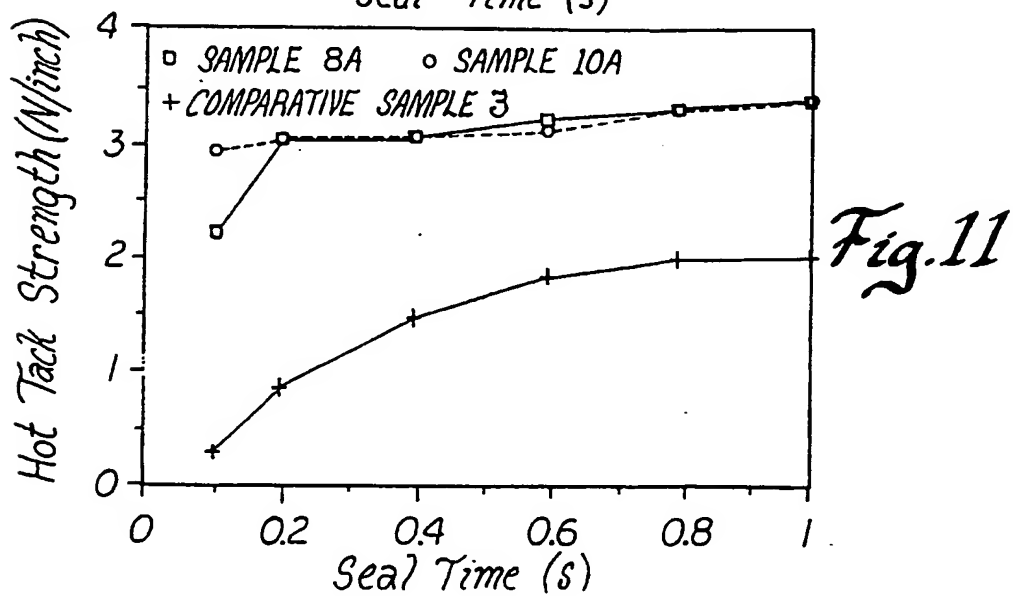
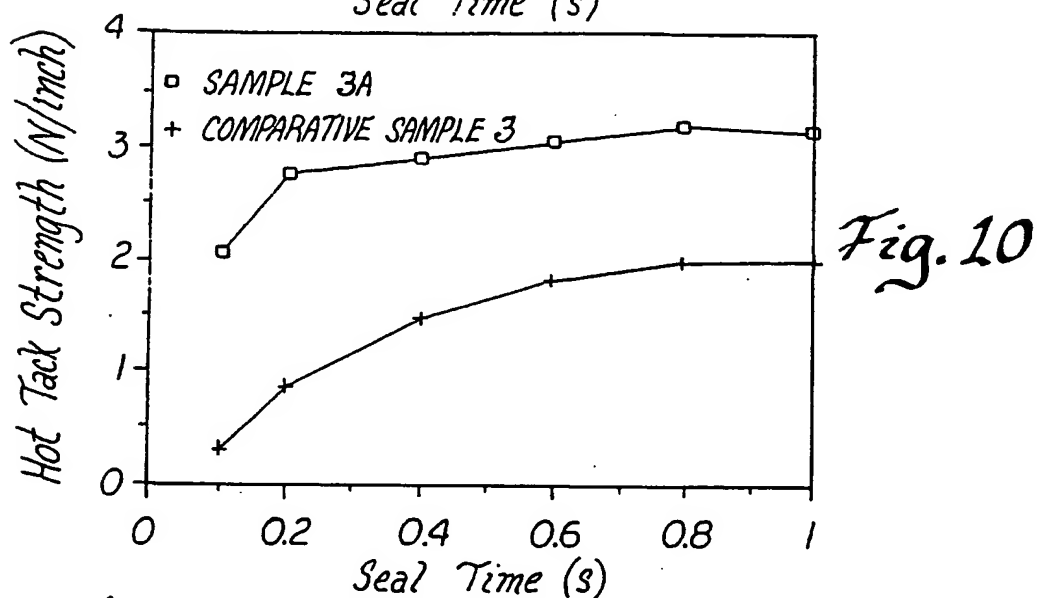
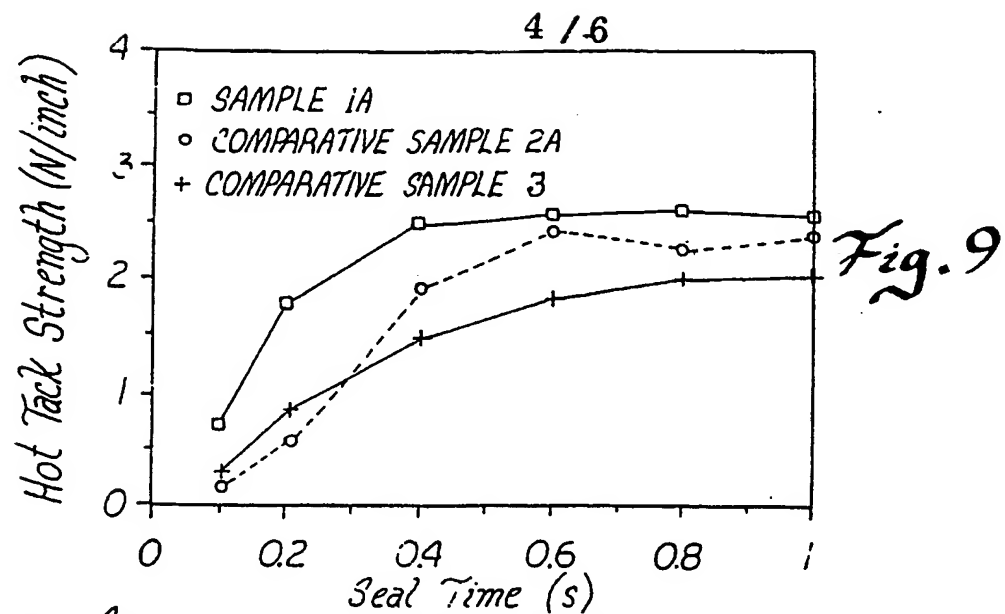


Fig. 8



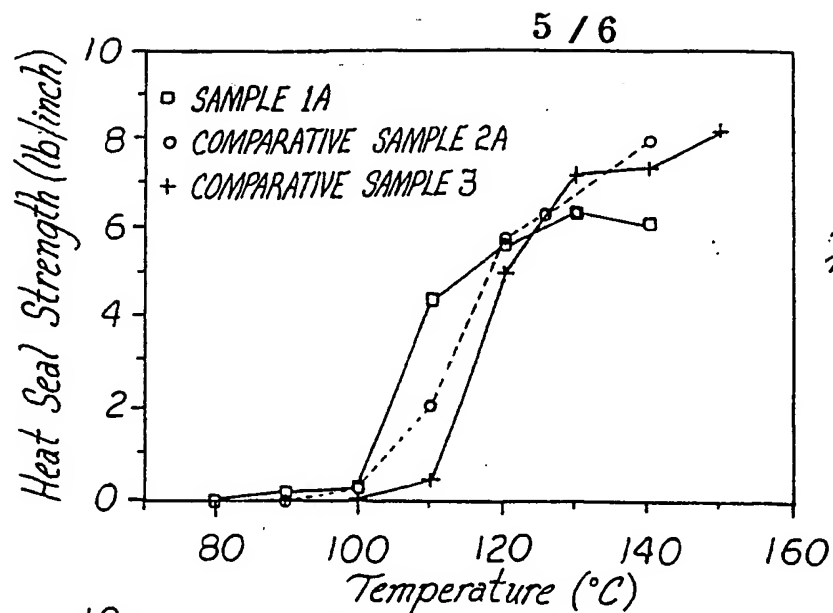


Fig. 12

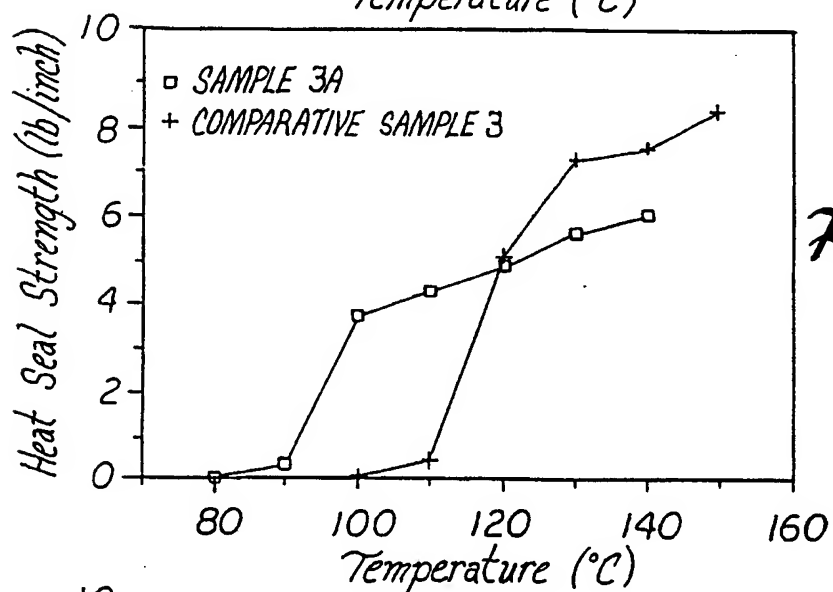


Fig. 13

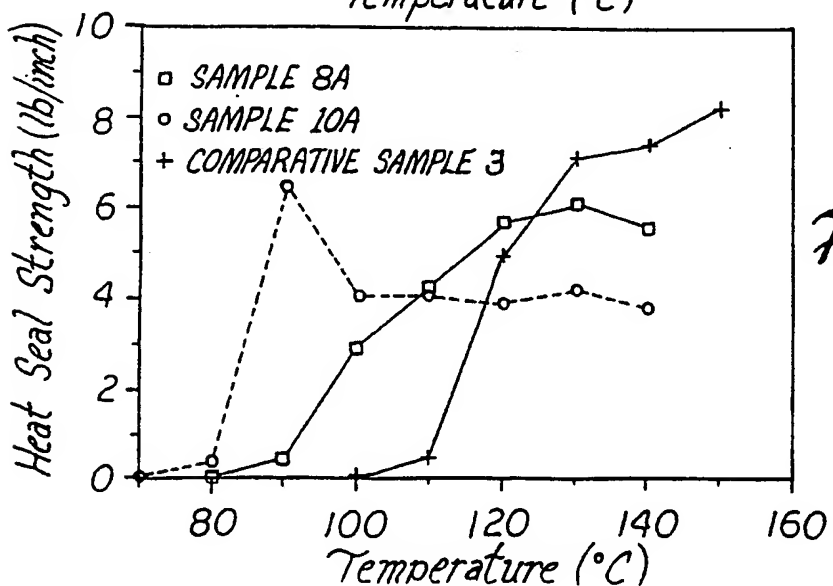


Fig. 14

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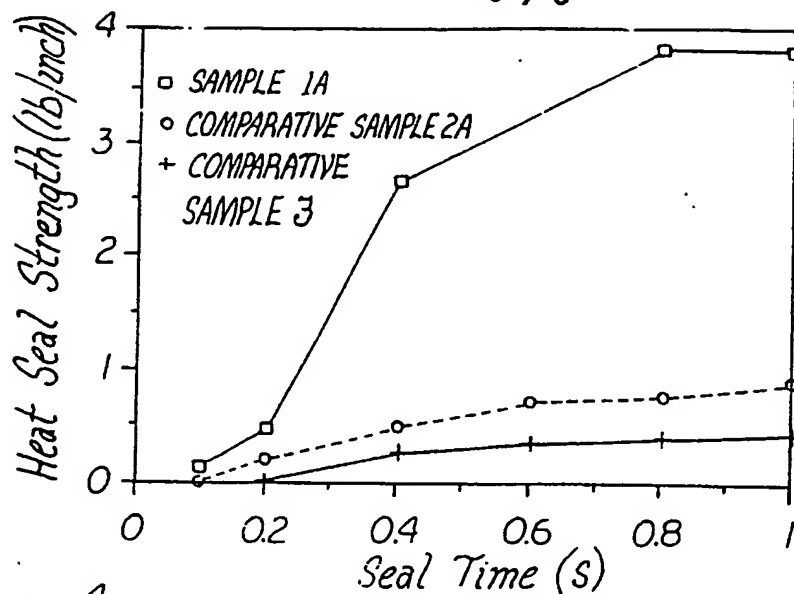


Fig. 15

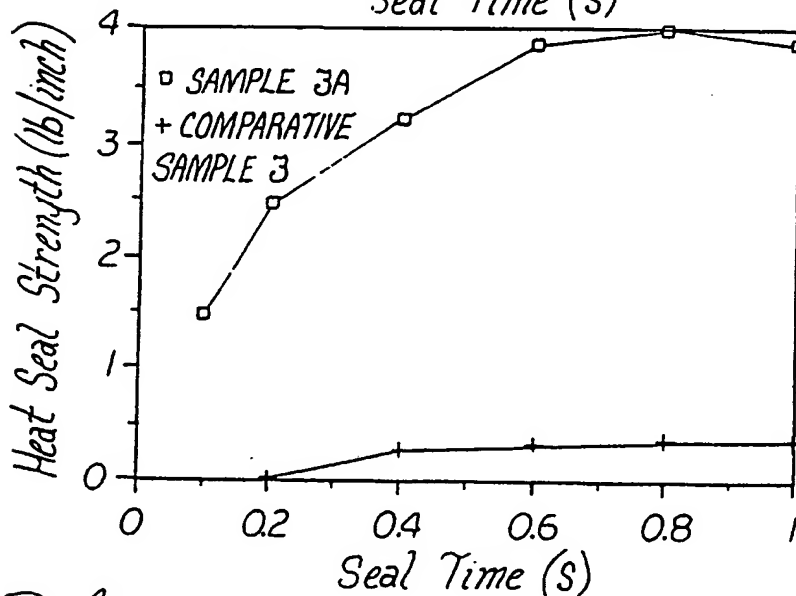


Fig. 16

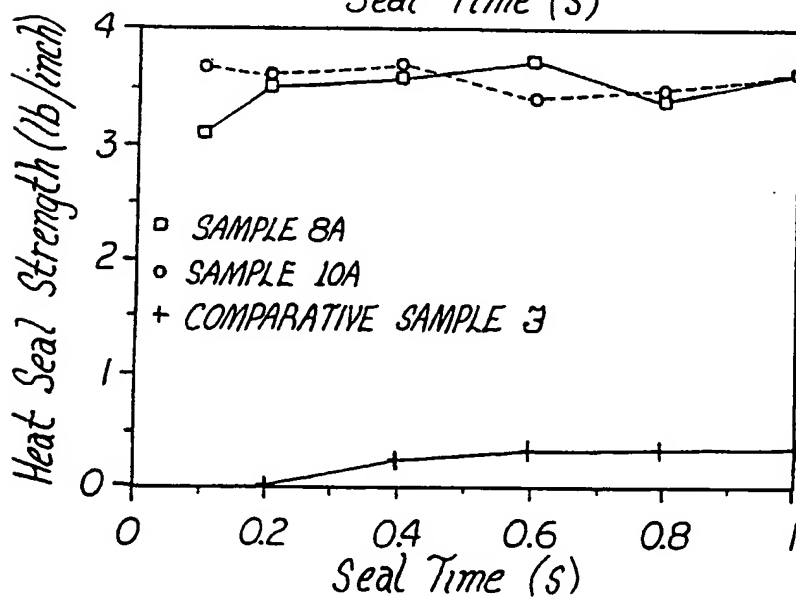



Fig. 17

I. CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all) ⁶		
According to International Patent Classification (IPC) or to both-National Classification and IPC		
Int.Cl. 5 B32B27/08; C09J123/04; B65D65/40		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁷		
Classification System	Classification Symbols	
Int.Cl. 5	B32B ; C09J ; C08L	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸		
III. DOCUMENTS CONSIDERED TO BE RELEVANT⁹		
Category ¹⁰	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
X	EP,A,0 057 238 (MITSUI PETROCHEMICAL INDUSTRIES) 11 August 1982 see claims 1,4,7 see page 11, line 10 - line 32 see page 14, line 17 see page 9, line 29 - page 10, line 17 ---	1-11
Y	GB,A,2 121 062 (DU PONT CANADA INC.) 14 December 1983 see the whole document ---	1,5,6,8, 11
Y	EP,A,0 221 726 (MOBIL OIL CORPORATION) 13 May 1987 see the whole document ---	1,5,6,8, 11
A	EP,A,0 305 959 (VISKASE CORPORATION) 8 March 1989 see claim 17 ---	1
-/--		
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IV. CERTIFICATION		
Date of the Actual Completion of the International Search 28 OCTOBER 1992		Date of Mailing of this International Search Report 5. 11. 92
International Searching Authority EUROPEAN PATENT OFFICE		Signature of Authorized Officer DE JONGE S.J.P. 

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.
A	US,A,4 801 486 (QUACQUARELLA ET AL) 31 January 1989 see claims -----	1

**ANNEX TO THE INTERNATIONAL SEARCH REPORT
ON INTERNATIONAL PATENT APPLICATION NO.**

US 9206582
SA 63437

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on
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